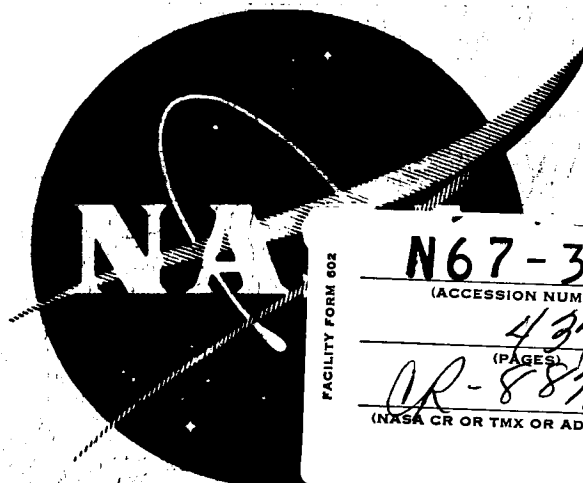


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Potassium Corrosion Test Loop Development Topical Report No. 2

MATERIAL AND PROCESS SPECIFICATIONS FOR REFRACTORY ALLOY AND ALKALI METALS

By

R. G. Frank, D. N. Miketta, W. H. Kearns,
W. R. Young, and R. B. Hand

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION

GENERAL ELECTRIC

CINCINNATI, OHIO 45215

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Contract NAS 3-2547

December 13, 1965

Technical Management
NASA-Lewis Research Center
Space Power Systems Division
T. A. Moss and R. L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
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FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2547. The purpose was the preparation of comprehensive material and process specifications for the procurement, cleaning, and joining of refractory alloys and for the procurement, purification, and handling of the alkali metals. The refractory alloys and alkali metals are to be used in the construction and evaluation of a boiling and condensing potassium corrosion test loop. Although the specifications found herein contain the General Electric Company's designation, anyone who so desires is encouraged to use the contents of the prepared specifications and assign their own specification numbers.

The refractory alloy material and process specifications were prepared by Mr. D. N. Miketta (deceased) under the direction of Mr. R. G. Frank, Manager, Physical Metallurgy. Specifications for joining were prepared by Mr. W. H. Kearns and Mr. W. R. Young, Manager, Joining and Fabrication. Alkali metal specifications were prepared by Dr. R. B. Hand, Manager, Chemistry and Physics. This work was administered for the General Electric Company by Dr. J. W. Semmel, Jr., Manager, Materials and Processes. Mr. E. E. Hoffman, Manager, Corrosion Technology, acts as Program Manager of the Potassium Corrosion Test Loop Development Program which will evaluate the material performance. Messieurs T. A. Moss and R. L. Davies are the Technical Managers for the National Aeronautics and Space Administration.

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I. INTRODUCTION

A forced circulation, two phase boiling and condensing potassium loop testing program is being conducted under NASA Contract NAS 3-2547 to develop a Prototype Corrosion Test Loop for the evaluation of advanced refractory containment and turbine alloys in environments which simulate projected space electric power systems. The Prototype Corrosion Loop consists of a two-loop Cb-1Zr alloy facility; sodium is heated by direct resistance in the primary loop and is used in a heat exchanger to boil potassium in the secondary corrosion test loop. Heat rejection for condensation in the secondary loop is accomplished by radiation in a high-vacuum environment. The corrosion test design conditions for the Prototype Loop are shown below; however, it is expected that the temperatures could be increased by about 400°F when testing is extended to include advanced refractory containment alloys that are stronger than Cb-1Zr alloy:

1. Boiling temperature - 1900°F
2. Superheat temperature - 2000°F
3. Condensing temperature - 1350°F
4. Subcooling temperature - 800°F
5. Mass flow rate - 20 to 40 lbs/hr
6. Boiler exit vapor velocity - 100 to 150 ft/sec
7. Average heat flux in the boiler - 50,000 to 100,000 BTU/hr ft²

Prior to the construction and operation of the Prototype Corrosion Test Loop, two other Cb-1Zr alloy test loops were constructed and operated in a series of component evaluation and endurance tests. Loop I, a natural convection loop, was operated for 1000 hours with liquid sodium at a maximum temperature of 2250° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, flowmeter, flow control and isolation valves, and pressure transducers has completed 2650 hours of scheduled testing at a pump inlet temperature of 1985°F.

This report covers the preparation of material and process specifications for the procurement, cleaning, and joining of refractory alloys and for the procurement, purification, and handling of the alkali metals to be used in the fabrication and evaluation of the test loops. The major objective in the preparation of the specifications was to assure the utilization of only high quality products with uniform and reproducible properties.

II. PROCUREMENT OF REFRACTORY ALLOY MILL PRODUCTS

A. Material Specifications

The material specifications that were prepared and utilized for the procurement of refractory metals and alloys for Tasks I-IV of the Potassium Corrosion Test Loop Development Program are listed in Table I and are reproduced in Appendix A. By far the greatest bulk of material required for the construction of Loops I and II and the Prototype Loop was bar and rod, sheet, plate and foil, and seamless tubing and pipe of Cb-1Zr alloy. Small quantities of Mo-TZM alloy bar for the turbine simulator and valves and T-111 alloy bar and sheet for the fast response transducer were required. In addition, trial orders for small quantities of thin wall seamless tubing of T-111, T-222, and D-43 alloy were placed to determine the feasibility of fabricating bellows from the higher strength columbium and tantalum alloys. Preparation of the specifications for T-111, T-222, and D-43 alloys are discussed elsewhere (1). Subsequent to issuing the material specifications, a revision was made in the specification numbering system; both the revised and original specification numbers are shown in Table I and on the cover sheet of each specification in Appendix A.

Generally, it was found that the inclusion of detailed processing procedures in the specifications for Cb-1Zr alloy was too restrictive, particularly in the production of large bar and tube hollows. Assurance of obtaining the desired quality product can be achieved by establishing more stringent requirements in other sections of the specifications, i.e., stress-rupture properties. Also, it was found desirable to establish specifications for applications of varying requirements, i.e., structural or nonstructural. As a result, the specifications for Cb-1Zr alloy mill products have been revised and re-issued. The revised specifications for Cb-1Zr alloy are listed in Table II and reproduced in Appendix B.

It can be concluded that the existence of fairly stringent specifications contributed significantly to the successful procurement of generally high quality mill products.

B. Vendor Selection

The procurement of the refractory alloy mill products for the program was initiated by personal visits by General Electric personnel to vendors representative of the industry and considered capable of supplying the program's material needs. The purpose of these visits were: 1) to review vendor facilities and previous experience in producing the desired mill products of the alloys of interest,

(1) Miketta, D. N., and Frank, R. G., "Potassium Corrosion Test Loop Development," October 1, 1965, NASA Contract NAS 3-2547, NASA-CR-54761.

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i.e., primarily Cb-1Zr alloy, 2) to present the program's technical requirements and material delivery schedules, and 3) to review the NASA-Lewis quality assurance program provisions for research, test, and development programs (QA-2a). The vendors contacted were:

1. Wah Chang Corporation, Albany, Oregon
2. Kawecki Chemical Company, Boyertown, Pennsylvania
3. Stellite Division, UCC, Kokomo, Indiana
4. Superior Tube, Norristown, Pennsylvania
5. Stauffer Metals Division, Richmond, California

Formal inquiries to supply Cb-1Zr alloy mill products were prepared and submitted to the vendors for quotation. The inquiries covered the entire scope of standard mill products ranging from foil, sheet, plate, and bar to pipe and tubing of various diameters and wall thicknesses. The General Electric Company, Space Power and Propulsion Section specifications 01-0003-02-B, "Bar, Rod, Sheet, Plate and Strip: Columbium-1% Zirconium" and 01-0004-02-P, "Seamless Tubing: Columbium-1% Zirconium Alloy" formed the basis around which the inquiries were framed. Invitations to bid on the entire Cb-1Zr alloy requirements were offered to Wah Chang Corporation, Kawecki Chemical Company and Stauffer Metals Division. Vendors whose stated interests were limited to specific mill products were given the opportunity to quote on those items within their realm of activity, i.e., Superior Tube Company, Wolverine Tube Division, and Bishop Tube Company for hollow products and Stellite Division, UCC, E. I. DuPont de Nemours, and Fansteel Metallurgical Corporation for mill products of solid cross section.

The quotations were carefully reviewed and composite tabulations were prepared for purposes of comparison with emphasis of the final vendor selection being placed on adherence to the specification, price, delivery, and previous experience with that vendor on past programs. In general, adherence to the requirements of the specifications was satisfactory. The greatest and most repeated areas of concern on the part of the vendors arose from the number of tests required, the stress-rupture requirements, and the defect level and techniques specified in the ultrasonic inspection section of the specifications. Mutual agreements on minor modifications to the specifications were reached in all cases. Several vendors who could not fulfill the ultrasonic inspection requirements of the specifications were persuaded to enlist the services of outside testing sources specializing in that type of inspection. Where invocation of the overall specification was impractical and unnecessary, i.e., wire and foil, only those paragraphs of the specification dealing with chemistry, particularly interstitial levels, and general soundness and cleanliness were specified.

One product, the 0.008-inch thick wall tubing of Cb-1Zr alloy, had to be procured on an "aim for" basis because of a lack of background data and experience to ensure the vendor of meeting the specification. The resulting product was

higher in oxygen content (868 ppm) than desired, and although satisfactory bellows were fabricated from the product, it was appropriate to determine whether 0.008-inch thick wall tubing could be obtained within the basic specification (300 ppm oxygen maximum) from another source. A trial order on an "aim for" basis to specification 01-0004-02-B was placed for 50 pieces of Cb-1Zr alloy 0.375-inch OD x 0.008-inch thick wall x 7-inch long tubing from Wolverine Tube Division. At the same time a similar order was placed with Wolverine Tube Division for D-43 alloy tubing to determine the feasibility of processing thin wall tubing of an advanced high strength columbium alloy. As was expected, utilization of the vacuum annealing facilities at Wolverine Tube Division resulted in the production of thin wall, columbium alloy tubing having an oxygen content below 350 ppm.

The small quantity of Mo-TZM bar for the turbine simulator and for the valve components were purchased from Climax Molybdenum Company to their specification (CMX-WB-TZM-1). The T-111 tantalum alloy bar and foil were ordered from National Research Corporation (NRC) to purchase order type specification. It also was deemed desirable to obtain preliminary experience in the production of thin-walled, high strength tantalum alloy tubing. Purchase orders were placed with National Research Corporation for 50 pieces each of 0.375-inch OD x 0.008-inch thick wall x 7-inch long tubing of T-111 and T-222 alloys to General Electric specifications 01-0035-00-B and 01-0036-00-C.

Table III summarizes the refractory alloy requirements for the Potassium Corrosion Test Loop Development Program and the final selection of the vendors and the particular products each vendor supplied for the program.

C. Quality Assurance

The quality assurance program was established to provide adequate identification and documentation of the quality of the refractory alloys used in the construction of the test loops. In addition, the information acquired in the quality assurance program would fulfill the material documentation required for the test history of the loop components.

The majority of the quality assurance measures were performed and certified to be within specification by the materials producers and check tests performed by the General Electric Company generally were limited to chemical analyses of the interstitial elements, metallographic examination, hardness measurements, and visual inspection of the incoming products.

Upon receipt of material from the material producers, a Material Control Number (MCN) was assigned to each homogeneous lot of material. A homogeneous lot included all material of the same size, shape, condition, and finish from one heat of material and which had received the same processing, had been annealed in the same vacuum annealing charge, and had been processed in the same manner in all operations in which the processing temperatures exceeded 500°F. Control of the assigned Material Control Numbers, Quality Assurance Test Records, and Material Disposition was aided through the appropriate use of the record forms shown in Appendix C.

A listing of the refractory alloy mill products procured for the program, the specifications to which they were procured, and the results of the quality assurance tests is presented in Tables IV through VII. A summary of the quality assurance test results with respect to meeting the requirements of the specifications is shown in Table VIII.

In general, it was evident that careful attention was given to the production of the mill products by the material producers. The only discordant note in visual inspection was found in the Cb-1Zr alloy tubing items; many tubes sustained evidence of scratches and nicks on the outside diameters as a result of poor handling and packing methods employed by the vendor. With the exception of the 0.008-inch thick wall tubing received from one vendor (MCN 410) and two lots of 0.002-inch foil (MCN 401 and 482), all the Cb-1Zr alloy mill products received have satisfied the interstitial element product analysis requirement of the specifications. The 0.008-inch wall tubing was higher in oxygen and carbon content than desired and the 0.002-inch thick foil was high in carbon content. However, in some instances, there was considerable deviation from the analyses supplied by the vendors and those obtained at General Electric. These variations attest to the continuing reluctance expressed by most vendors in having a two source check analysis of interstitial levels as a basis for rejection of refractory metal products. A summary of the variation in interstitial element content with the section size of the smaller Cb-1Zr alloy mill products is given in Table IX.

All the Cb-1Zr alloy mill products met the room temperature tensile and hardness requirements of the specifications and of those items tested for stress-rupture strength, one item, the 2-inch diameter bar (MCN 425), failed to meet the requirements. The grain size of all bar, sheet, plate, and tube with a wall thickness greater than 0.065 inch was well within the specification limits. The microstructure of a 0.375-inch OD x 0.065-inch thick wall tube is shown in Figure 1. Although the thin wall tubing, 0.008 inch and 0.025 inch, failed to meet the general grain size specification, the limits for thin wall tubing were over restrictive from a production standpoint and the grain size specification subsequently has been relaxed from the required 30 grain boundary intersections across the wall thickness to a maximum ASTM standard grain size number of 6. The two items of tubing that failed to meet the original specification would meet this requirement. The microstructure of a 0.375-inch OD x 0.008-inch thick wall tube is shown in Figure 2. All tubing items passed the hydrostatic and flare test requirements.

Nondestructive testing of Cb-1Zr alloy mill products for surface imperfections and internal defects generally found the material to be of high quality, except that a few surface tears were found on the 0.500-inch diameter bar, the surface of some of the tubing was rough, and six 0.375-inch OD x 0.065-inch thick wall tubes and three of the 1.0-inch OD x 0.100-inch thick wall tubes were rejected for internal defects greater than 3% of the wall thickness. However, in all cases it was possible to remove the identified defective areas and utilize the balance of the tube in the construction of the loops. A typical ID defect in a 0.375-inch OD x 0.065-inch thick wall Cb-1Zr alloy tube is shown in Figure 3.

Examination of the quality assurance tests performed on other refractory metals and alloys utilized in the program will reveal that the unalloyed tantalum and Mo-TZM alloy mill products met all the specified requirements. The microstructure of the Mo-TZM alloy one-inch diameter bar used for the machining of the blades in the turbine simulator is shown in Figure 4. However, all four items of the T-111 alloy failed to meet specifications: the hafnium was below the 2.0% minimum on three items; carbon was above the 30 ppm maximum on three items; tensile elongation was below the 15% minimum on one item; and major ultrasonic defects were found in the one-inch diameter bar.

Of the trial orders placed for 0.375-inch OD x 0.008-inch thick wall tubing of Cb-1Zr, D-43, T-111 and T-222 alloys, acceptable tubing was received from Cb-1Zr and T-111 alloys with the exception of carbon content. However, there was considerable disagreement between the vendor and General Electric analyses of the carbon in the Cb-1Zr alloy tubing, i.e., 79 ppm vs 145 ppm, respectively, with the specification being 100 ppm maximum. Regardless, a considerable reduction in interstitial element content was realized in the Cb-1Zr alloy tubing received from Wolverine Tube Division:

<u>Vendor</u>	<u>Chemical Analysis, ppm</u>			
	<u>C</u>	<u>O</u>	<u>N</u>	<u>H</u>
Wolverine Tube Division	79/145	260/347	35/8	7/8
Superior Tube Company	210/150	660/827	72/106	9/1

The microstructure of the Cb-1Zr alloy tubing received from Wolverine is shown in Figure 5.

The carbon content of T-111 alloy tubing was significantly higher than the specification (75 ppm maximum) as analyzed by the vendor and General Electric, i.e., 197 ppm and 225 ppm, respectively. The microstructure of the T-111 alloy is shown in Figure 6.

No tubing was received of T-222 alloy due to the inability of the vendor to produce the material; the D-43 alloy tubing generally was of poor quality with respect to surface condition, Figure 7.

III. MATERIAL CLEANING AND JOINING

The processing specifications utilized in the fabrication of Loop I, Loop II, and the Prototype Loop for the Potassium Corrosion Test Loop Development Program are listed in Table X and are reproduced in Appendix D. In general, these specifications provided excellent guidelines for the processes involved.

Certain technological advances made during the past two years have provided information which will be incorporated into future specifications to be used for the fabrication of a T-111 tantalum alloy loop under NASA Contract NAS 3-6474. The welding specification for Cb-1Zr alloy, 03-0005-00-A, did not require monitoring of gaseous impurities within the welding chamber. However, as described in Topical Report No. 1 (2), procedures have been developed for monitoring the helium within the welding chamber. Limits on the levels of oxygen, nitrogen, and water vapor in the welding chamber will therefore be incorporated into revised welding specifications for refractory metals and their alloys. The inclusion of the postweld annealing requirements within the body of the welding specification presented some difficulties in quality control documentation due to the fact that a substantial number of welds were usually annealed in one furnace run. Therefore, the postweld annealing operation will be incorporated into a separate specification for future work. Also, it was found that many commercially available vacuum furnaces could not maintain the specified 1×10^{-5} torr pressure at the 2200°F annealing temperature. This vacuum requirement will be retained in future specifications since the technical requirements have not changed. The specification will place additional emphasis on furnace qualifications by chemical analysis of samples and on the use of protective refractory alloy foils for wrapping components.

The chemical cleaning specification, 03-0010-00-B, was implemented without difficulty after preliminary adjustment of the acid cleaning solution temperature which was specified to be 100°-125°F. At these bath temperatures, heating of parts due to the relatively rapid chemical reaction resulted in the formation of a white film during transfer to the water rinse tank. This problem was eliminated during the cleaning of loop components by maintaining the acid cleaning bath at room temperature.

Handling procedures which were used during assembly were defined on manufacturing instructions. These instructions were designed to provide both the processing sequence information and the requirements for cleanliness control. In future work, a separate specification will be prepared for cleanliness control during processing, i.e., forming, welding, and assembly operations, in order to simplify the quality control documentation.

The other specifications presented no obvious deficiencies and will be used for future work with minor modifications.

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- (2) Lyon, T. F., Potassium Corrosion Test Loop Development Topical Report No. 1, "Purification and Analysis of Helium for the Welding Chamber," July 1, 1965, NASA Contract NAS 3-2547, NASA-CR-54168.

IV. PROCUREMENT, PURIFICATION, AND HANDLING OF ALKALI METALS

Four specifications for the procurement and purification of high purity alkali metals were prepared for utilization in the program. These specifications are listed in Table XI and are reproduced in Appendix E. Included in the specifications reproduced in Appendix D are minor changes in the maximum allowable impurities that were made in May 1965. In the case of both sodium and potassium, the maximum allowable limits for some impurities were increased to coincide with the precision limitations of the emission spectrographic techniques currently in use. These maxima have been increased from 1 ppm to 5 ppm.

High purity reactor grade sodium for the program was procured from U.S. Industrial Chemicals Corporation (USI), Ashtabula, Ohio. Although USI took exception to specification 01-0031-00-B with respect to the maxima specified for impurities, only once have they failed in a minor way to meet the limits in the specification. In this instance they obtained a value of 63 ppm oxygen instead of the specified 50 ppm. The Mine Safety Appliance Research Corporation, Callery, Pennsylvania, would also supply sodium to specification 01-0031-00-B, again with several exceptions with regard to the maximum allowable impurity limits and the number of elements specified. Other vendors contacted either refused to quote or took exception to the specification on the basis that the cost would be excessive to determine whether or not they could meet the specification, particularly with regard to developing appropriate analytical methods.

Certain impurity elements of interest in sodium are listed in Table II of specification 01-0031-00-B, with the maximum allowable concentration limits designated as "unspecified". Vendors normally do not analyze for these elements and, consequently, have no prior knowledge on which to base limits for their product. However, analyses of as-received sodium that were conducted for the General Electric Company by the Nuclear Materials Equipment Corporation have generally shown concentrations of less than 10 ppm each for these elements.

The only vendor who would comply with the specification for high purity potassium, 01-0033-00-B, was Mine Safety Appliance Research Corporation (MSA). All potassium that was procured for the program was obtained from MSA. MSA will guarantee all sections of the specification and, in addition, will guarantee a carbon content of less than 50 ppm.

Overall, it is believed that the specifications for the procurement of high purity sodium and potassium are realistic and no changes are planned in their content at this time. However, both specifications should be revised in the future in order to eliminate or modify the requirement that sodium should assay 99.95% and potassium, 99.99%. It is obvious that if all impurities present were at the allowable maxima these requirements could not be met. It is intended to place a maximum on the sum of the concentrations of all elements for which analyses are made.

Specifications 01-0032-00-B, "Hot Trapped Reactor Grade Sodium Metal", and 01-0034-00-B, "Hot Trapped High Purity Grade Potassium Metal", also were revised in May 1965. Included in the revisions were the up-dating of the referenced specification numbers, increasing the maximum allowable limits of cobalt, boron, silver, and molybdenum from 1 ppm to 5 ppm and reducing the requirement for the minimum hot trapping duration from 100 to 200 hours. This latter change was made because the results obtained from more recent purification operations have shown that extended periods of hot trapping are not necessary when high purity starting materials are used. The construction of hot traps and the hot trapping procedures have conformed to those specifications, and the alkali metals obtained have met the purity requirements. There are no reasons, at present, to further modify these specifications.

Specification 03-0018-00-A, "Alkali Metal Handling and Control Procedures", contains the instructions for sampling and analysis referred to in the hot trapping specifications. This comprehensive set of instructions requires periodic revision to take into account advances in the alkali metal technology. With respect to current practice at this laboratory, the specification is quite up-to-date in its present form, but it will be extended in the next few months to include newly established procedures for the determination of nitrogen in lithium and metallic elements in sodium and lithium, as required for future work.

TABLE I. REFRACTORY ALLOY MATERIAL SPECIFICATIONS

Title	Revised Specification No.	Original Specification No.
1. Bar, Rod, Sheet, Plate and Strip: Columbium-1% Zirconium Alloy	01-0003-02-B	SPPS-1B Amend.
2. Bar, Rod, Sheet, Plate and Strip: Columbium-1% Zirconium Alloy	01-0003-03-B	---
3. Bar, Rod, Sheet, Plate and Strip: Columbium-1% Zirconium Alloy	01-0003-04-B	---
4. Seamless Tubing: Columbium-1% Zirconium Alloy	01-0004-02-B	SPPS-2B Amend.
5. Seamless Tubing: Columbium-1% Zirconium Alloy	01-0004-03-B	---
6. Bar and Rod: Mo-TZM (Mo-0.5Ti-0.08Zr) Alloy	01-0009-00-B	SPPS-15-R1*
7. Bar and Rod: Mo-TZM (Mo-0.5Ti-0.08Zr) Alloy	01-0009-01-B	---
8. Climet TZM (Mo-0.5%Ti-0.08%Zr) Wrought Bar	CMX-WB-TZM-2	CMX-WB-TZM-1
9. Bar and Rod: T-111 (Ta-8W-2Hf) Alloy	01-0015-00-B	SPPS-22-R1*
10. Seamless Tubing and Pipe: T-111 (Ta-8W-2Hf) Alloy	01-0035-00-B	SPPS-54-R1*
11. Sheet, Plate and Strip: T-111 (Ta-8W-2Hf) Alloy	01-0040-00-B	SPPS-59-R1*
12. Seamless Tubing and Pipe D-43 (Cb-10W-1Zr-0.1C) Alloy	01-0037-00-B	SPPS-56-R1*
13. Seamless Tubing and Pipe: T-222 (Ta-10.4W-2.4Hf-0.01C)	01-0036-00-C	SPPS-55-R2*
14. Tantalum Ingots and Flat Mill Products	ASTM B-364-62T	ASTM B364-61T*
15. Tantalum Rod and Wire	ASTM B-365-62T	ASTM B-365-61T*

* Modified by Purchase Order.

TABLE II. REVISED MATERIAL SPECIFICATION FOR Cb-1Zr ALLOY

<u>Title</u>	<u>Specification Number</u>
Seamless Tubing and Pipe: Columbium-1% Zirconium Alloy	01-0004-00-D
Seamless Tubing and Pipe: Columbium-1% Zirconium Alloy	01-0004-01-D
Bar and Rod: Columbium-1% Zirconium Alloy	01-0052-00-B
Bar and Rod: Columbium-1% Zirconium Alloy	01-0052-01-B
Sheet, Plate and Strip: Columbium-1% Zirconium Alloy	01-0053-00-B
Sheet, Plate and Strip: Columbium-1% Zirconium Alloy	01-0053-01-B
Foil: Columbium-1% Zirconium Alloy	01-0054-00-A
Wire: Columbium-1% Zirconium Alloy	01-0055-00-A

TABLE III. SUMMARY OF REFRACTORY ALLOY MILL PRODUCT REQUIREMENTS

Material	Vendor	Mill Product		
		Form	Size, Inch	Weight, lbs.
Cb-1Zr	Kawecki Chemical Company	Foil	0.002 x 3.5	7.0
	Kawecki Chemical Company	Foil	0.002 x 0.5	44.7
	Kawecki Chemical Company	Foil	0.005 x 3.0	0.4
				52.1
	Fansteel	Sheet	0.0175 x 12	3.2
	Wah Chang	Sheet	0.0175 x 12	6.1
	Wah Chang	Sheet	0.030 x 12	8.1
	Wah Chang	Sheet	0.032 x 12.5	23.3
	Wah Chang	Sheet	0.032 x 10.5	6.3
	Wah Chang	Sheet	0.032 x 12	14.7
	Wah Chang	Sheet	0.125 x 10	68.2
	Wah Chang	Sheet	0.125 x 30	88.7
				218.6
	Wah Chang	Plate	0.250 x 12	48.2
	Wah Chang	Plate	0.50 x 6	113.5
	Stellite	Plate	0.25 x 5	62.6
				224.3

TABLE III. (Cont'd)

<u>Material</u>	<u>Vendor</u>	<u>Mill Product</u>		
		<u>Form</u>	<u>Size, Inch</u>	<u>Weight, lbs</u>
Cb-1Zr	Kawecki Chemical Company	Wire	0.20 diameter	0.3
	Stellite	Wire	0.062 diameter	12.7
	Stellite	Wire	0.094 diameter	12.7
				25.7
	Stellite	Bar	0.125 diameter	16.6
	Stellite	Bar	0.375 diameter	1.7
	Stellite	Bar	0.50 diameter	8.1
	Stellite	Bar	1.0 diameter	8.9
	Stellite	Bar	1.5 diameter	26.9
	Stellite	Bar	2.0 diameter	59.0
	Stellite	Bar	0.5 x 1.0	21.3
	Stellite	Bar	1.0 x 1.0	10.4
	Fansteel	Bar	0.375 diameter	0.6
	Fansteel	Bar	0.500 diameter	2.2
	Fansteel	Bar	0.750 diameter	1.6
	Fansteel	Bar	1.125 diameter	1.8

TABLE III. (Cont'd)

Material	Vendor	Mill Product		
		Form	Size, Inch	Weight, lbs
Cb-1Zr	Fansteel	Bar	1.250 diameter	9.1
	Fansteel	Bar	2.00 diameter	6.0
	Fansteel	Bar	2.50 diameter	27.8
	Fansteel	Bar	3.125 diameter	41.1
	Wah Chang	Bar	0.50 diameter	6.0
	Stauffer	Bar	2.50 diameter	60.0
	Stauffer	Bar	3.00 diameter	76.0
	Stauffer	Bar	3.25 diameter	68.0
	Stauffer	Bar	3.50 diameter	81.2
				534.3
	Superior	Tube	0.375 OD x 0.008 wall	3.7
	Wolverine	Tube	0.375 OD x 0.008 wall	1.4
	Wah Chang	Tube	0.1875 OD x 0.025 wall	1.0
	Wah Chang	Tube	0.375 OD x 0.065 wall	48.0
	Wah Chang	Tube	0.25 OD x 0.062 wall	0.8
	Wah Chang	Tube	1.00 OD x 0.100 wall	34.5
	Wah Chang	Tube	2.75 OD x 0.125 wall	14.7

TABLE III. (Cont'd)

Material	Vendor	Mill Product		
		Form	Size, Inch	Weight, lbs
Cb-1Zr	Wah Chang	Tube	3.25 OD x 0.125 wall	13.8
	Reactive/Bishop	Tube	0.375 OD x 0.065 wall	19.7
	Kawecki Chemical Company	Pipe	0.375 Schedule 80	10.4
				148.0
D-43	Wolverine	Tube	0.375 OD x 0.008 wall	1.2
				1.2
Mo-TZM	Climax	Bar	0.125 diameter	0.1
	Climax	Bar	0.500 diameter	2.7
	Climax	Bar	0.875 diameter	5.2
	Climax	Bar	1.00 diameter	24.3
	Climax	Bar	2.00 diameter	69.4
				101.7
Ta	Wah Chang	Foil	0.003 x 6	10.0
	Wah Chang	Foil	0.002 x 8	8.9
				18.9
	Stellite	Sheet	0.032 x 0.75	0.2
	Stellite	Sheet	0.062 x 2.125	2.7
				2.9

TABLE III. (Cont'd)

<u>Material</u>	<u>Vendor</u>	<u>Mill Product</u>		
		<u>Form</u>	<u>Size, Inch</u>	<u>Weight, lbs</u>
Ta	Stellite	Wire	0.020 diameter	1.4
				1.4
		Bar	0.250 diameter	0.8
		Bar	0.625 diameter	2.2
	Stellite	Bar	1.125 diameter	4.9
				7.9
T-111	National Research Corporation	Tube	0.375 OD x 0.008 wall	1.9
				1.9
		Bar	0.1875 diameter	0.2
		Bar	1.0 diameter	5.7
				5.9
	National Research Corporation	Strip	0.015 x 1.5	0.6
	National Research Corporation	Strip	0.020 x 1.5	0.9
				1.5

TABLE IV. RESULTS OF QUALITY ASSURANCE TEST PROGRAM

Alloy	MCN Number	Mill Product		Vendor	Heat Number	Specifications		Meets All Specification Requirements	Remarks
		Form	Size			Number	Major Exceptions		
Cb-12r	400	Foil	0.002" x 3.5" x 125'	Kaweck Chemical Company	333-5	SPPS-1B	Chemistry only	No	Oily surface
	401-(1-29)	Foil	0.002" x 0.5" x 10,800'	Kaweck Chemical Company	333-5	SPPS-1B	Chemistry only	No	Oily surface; C above spec..
	402	Foil	0.005" x 3.0" x 8'	Kaweck Chemical Company	333-5	SPPS-1B	Chemistry only	Yes	
	481	Foil	0.002" x 3.5" x 125'	Kaweck Chemical Company	25908	SPPS-1B	Chemistry only	Yes	
	482	Foil	0.002" x 0.5" x 1350'	Kaweck Chemical Company		SPPS-1B	Chemistry only	No	C above spec.
	404-(1-12)	Tube	0.375" OD x 0.065" wall	Reactive/Bishop	11-228-01	SPPS-2B	Toll--best effort	Yes	ID surface rough; 4 of 12 tubes rejected on ultrasonic indications
	406	Tube Blank	2" OD x 0.25" wall x 4'	DuPont	11-228-01	SPPS-2B	Chemistry only	Yes	Used for MCN 404
	410-(1-204)	Tube	0.375" OD x 0.008" wall x 7" (204 pieces)	Superior Tube	355-70274	Best Effort	C 200 ppm Max. O 500 ppm N 300 ppm H 50 ppm	--	
	431-(1-6)	Tube	0.1875" OD x 0.025" wall	Wah Chang	355-70303	SPPS-2B	No stress-rupture	No	Grain size above maximum
	437-(1-22)	Tube	0.375" OD x 0.065" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture	Yes	
	438	Tube	2.75" OD x 0.125" wall	Wah Chang	5-53003	SPPS-2B	No stress-rupture	Yes	
	439	Tube	3.25" OD x 0.125" wall	Wah Chang	5-53003	SPPS-2B	No stress-rupture	Yes	
	440-(1-2)	Tube	0.375" OD x 0.065" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture; as-drawn	No	Major defect--ultrasonic
	441-(1-3)	Tube	1.0" OD x 0.100" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture; stress-relieved	No	Major defect--ultrasonic
	444	Tube	0.25" OD x 0.062" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture	Yes	
	445-(1-2)	Tube	0.375" OD x 0.065" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture	Yes	ID surface defect 445-1
	447	Tube	0.375" OD x 0.065" wall	Wah Chang	98-70546	SPPS-2B	No stress-rupture; as-drawn	Yes	
	466-(1-55)	Tube	0.375" OD x 0.008" wall	Wolverine Tube	11-052-1	Best Effort	----	--	
	467	Tube	0.375" OD x 0.008" wall x 5'	Wolverine Tube	11-052-1	Best Effort	----	--	
	470-(1-2)	Tube	0.675" OD x 0.126" wall x 78" (3/8 Sch. 80; 2 pcs.)	Kaweck Chemical Company	2590-P-4	SPPS-2B	No stress rupture	No	Ultrasonic indications 470-1
	405-(1-2)	Bar	2.5" diameter	Stauffer	FV-136	SPPS-1B	2300°F anneal	Yes	
	411-(1-2)	Bar	3.0" diameter	Stauffer	FV-136	SPPS-1B	2300°F anneal	Yes	
	412-(1-2)	Bar	3.25" diameter	Stauffer	FV-136	SPPS-1B	2300°F anneal	Yes	
	413	Bar	3.5" diameter	Stauffer	FV-136	SPPS-1B	2300°F anneal	Yes	
	419	Bar	0.125" diameter	Stellite	5120	SPPS-1B	None	Yes	
	420-(1-4)	Bar	0.375" diameter	Stellite	5155	SPPS-1B	None	Yes	
	421-(1-2)	Bar	0.500" diameter	Stellite	5155	SPPS-1B	None	Yes	
	422-(1-9)	Bar	0.500" diameter	Stellite	5120	SPPS-1B	None	Yes	
	423-(1-6)	Bar	1.0" diameter	Stellite	5155	SPPS-1B	None	Yes	
	424-(1-6)	Bar	1.5" diameter	Stellite	5155	SPPS-1B	None	Yes	Small surface tears

TABLE IV. (Cont'd)

Alloy	MCN Number	Mill Product		Weight	Vendor	Heat Number	Specifications		Meets All Specification Requirements	Remarks
		Form	Size				Number	Major Exemptions		
Cb-12r	425-(1-5)	Bar	2.0" diameter	59	Stellite	5154	SPPS-1B	None	No	SR life below minimum
	426-(1-20)	Bar	0.5" x 1.0"	21.3	Stellite	5155	SPPS-1B	None	Yes	
	427-(1-5)	Bar	1.0" x 1.0"	10.4	Stellite	5155	SPPS-1B	None	Yes	
	428-(1-7)	Bar	1.25" diameter	16.4	Stellite	5155	SPPS-1B	None	Yes	
	429-(1-3)	Wire	0.062" diameter	7.5	Stellite	5154	SPPS-1B	Chemistry only	Yes	
	430	Wire	0.094" diameter	7.5	Stellite	5154	SPPS-1B	Chemistry only	Yes	
	455	Wire	0.094" diameter	5.2	Stellite	5154	SPPS-1B	Chemistry only	Yes	
	456, 457, 458	Wire	0.062" diameter	5.2	Stellite	5154	SPPS-1B	Chemistry only	Yes	
	501	Wire	0.020" diameter x 250'	0.32	Kawachi Chemical Company	Ingot Number 27605-D	SPPS-1B	Chemistry only	Yes	
	478	Rod	1.250" diameter x 24"	9.1	Fansteel	80B849	SPPS-1B	No stress-rupture	Yes	
	479	Rod	0.5" diameter x 24"	1.5	Fansteel	80B849	SPPS-1B	No stress-rupture	Yes	
	483	Rod	0.375" diameter x 18"	0.61	Fansteel	80B971	SPPS-1B	No stress-rupture	No	
	484	Rod	0.5" diameter x 12"	0.73	Fansteel	80B971	SPPS-1B	No stress-rupture	Yes	
	485	Rod	0.750" diameter x 12"	1.64	Fansteel	80B971	SPPS-1B	No stress-rupture	Yes	
	486	Rod	1.125" diameter x 6"	1.8	Fansteel	80B971	SPPS-1B	No stress-rupture	Yes	
	488	Bar	2" diameter x 6"	6.0	Fansteel	2701	SPPS-1B	2400°F Anneal	Yes	
	499	Bar	2.5" diameter x 18"	27.8	Fansteel	2701	SPPS-1B	2400°F Anneal	Yes	
	500	Bar	3.125" diameter x 17"	41.1	Fansteel	2701	SPPS-1B	2400°F Anneal	Yes	
	510	Rod	0.5" diameter x 48"	6	Wah Chang	70559	SPPS-1B	No stress-rupture	Yes	
	407	Plate	0.250" x 12" x 50"	48.2	Wah Chang	911-70341	SPPS-1B	No stress-rupture	Yes	
	408-(1-3)	Plate	0.500" x 6" x 25"	72.5	Wah Chang	912-1211	SPPS-1B	No stress-rupture	Yes	
D-43	414	Plate	0.500" x 6" x 25"	23.4	Wah Chang	911-70341	SPPS-1B	No stress-rupture	Yes	H above spec.
	416-(1-2)	Plate	0.500" x 6" x 9"	17.6	Wah Chang	912-1018	SPPS-1B	No stress-rupture	Yes	
	468-(1-2)	Plate	0.250" x 5" x 72"	62.6	Stellite	5194	SPPS-1B	No stress-rupture	Yes	
	409-(1-4)	Sheet	0.0175" x 12" x 24"	6.1	Wah Chang	912-1211	SPPS-1B	No stress-rupture	Yes	
	415-(1-4)	Sheet	0.125" x 10" x 43"	68.2	Wah Chang	912-1018	SPPS-1B	No stress-rupture	Yes	
	417	Sheet	0.040" x 12" x 50"	7	Wah Chang	912-70112	SPPS-1B	No stress-rupture	Yes	
	418-(1-4)	Sheet	0.030" x 12" x 18"	8.1	Wah Chang	911-70341	SPPS-1B	No stress-rupture	Yes	
	432-(1-2)	Sheet	0.125" x 30" x 37"	88.7	Wah Chang	911-70341	SPPS-1B	No stress-rupture	Yes	
	454	Sheet	0.040" x 12" x 50"	7.7	Kawachi Chemical Company	373-342	SPPS-1B	Chemistry only	Yes	
	459	Sheet	0.032" x 10.5" x 60"	6.3	Wah Chang	912-1018	SPPS-1B	Chemistry, grain size only	Yes	
D-43	462-(1-2)	Sheet	0.032" x 12.5" x 60"	15.9	Wah Chang	912-1189	SPPS-1B	Chemistry, grain size only	Yes	---
	463	Sheet	0.032" x 12.5" x 60"	7.4	Wah Chang	912-1018	SPPS-1B	Chemistry, grain size only	Yes	
	497	Sheet	0.0175" x 12" x 24"	3.2	Fansteel	80B950	SPPS-1B	None	Yes	
	490	Tube	0.375" OD x 0.008" wall x 5'	1.2	Wolverine Tube	43-473	Best Effort	----	---	
	491-(1-35)	Tube	0.375" OD x 0.008" wall x 7'		Wolverine Tube	43-473	Best Effort	----	---	

TABLE IV. (Cont'd)

Alloy	MCN Number	Mill Product			Vendor	Heat Number	Specifications		Meets All Specification Requirements	Remarks
		Form	Size	Weight			Number	Major Exceptions		
D-43	492-(1-7)	Tube	0.375" OD x 0.008" wall x 7"	-	Wolverine	43-473	Best Effort	---	---	---
TZM	433	Bar	0.125" diameter	0.1	Climax	TZM 4-3934	CMX-WB-TZM-1	---	Yes	
	434	Bar	0.875" diameter	5.2	Climax	TZM 7473	CMX-WB-TZM-1	---	Yes	
	435-(1-2)	Bar	1.0" diameter	10.4	Climax	TZM 7474	CMX-WB-TZM-1	---	Yes	
	436	Bar	2.0" diameter	41.6	Climax	TZM 7501	CMX-WB-TZM-1	---	Yes	
	461	Bar	1.0" diameter	13.9	Climax	TZM 7479	CMX-WB-TZM-1	---	Yes	
	469	Bar	0.5" diameter	2.7	Climax	TZM 7436	CMX-WB-TZM-1	---	Yes	
	473	Bar	2.0" diameter	27.8	Climax	TZM 7555	CMX-WB-TZM-1	---	Yes	
	460	Foil	0.003" x 6"	10.0	Wah Chang	60165-Ta-53B	ASTM B364-61T	H < 10 ppm	Yes	
	477	Foil	0.002" x 8" x 620"	5.9	Wah Chang	60165-Ta-53B	ASTM-B364-62T	H < 15 ppm	Yes	
	480	Foil	0.002" x 8" x 315"	2.98	Wah Chang	61029N-Ta	ASTM-B364-62T	H < 15 ppm	Yes	
T-111	471	Sheet	0.062" x 2.125" x 32"	2.70	Stellite	TN-133	ASTM-B364-61T	H < 15 ppm	Yes	
	472	Sheet	0.032" x 0.750" x 12"	0.20	Stellite	EB-80	ASTM-B364-61T	H < 15 ppm	Yes	
	474	Rod	0.250" diameter x 24"	0.75	Stellite	81159	ASTM-B366-61T	H < 15 ppm	Yes	
	475	Rod	0.625" diameter x 12"	2.20	Stellite	81167	ASTM-B365-61T	H < 15 ppm	Yes	
	476	Bar	1.125" diameter x 8"	4.90	Stellite	81166	ASTM-B365-61T	H < 15 ppm	Yes	
	483	Wire	0.020" diameter x 7200"	1.40	Stellite	81159	ASTM-B365-61T	H < 15 ppm	Yes	
	448	Bar	0.1875" diameter x 12"	0.2	NRC	2650	P.O. Chemistry, Tensile, Elongation, Grain size	---	No	Hf below spec.
	449	Bar	1.0" diameter x 12"	5.7	NRC	3171	P.O. Chemistry, Tensile, Elongation, Grain size	---	No	Major defects--ultrasonic; C above spec.
	450	Strip	0.015" x 1.5" x 48"	0.6	NRC	2691	P.O. Chemistry, Tensile, Elongation, Grain size, Bend	---	No	Tensile elongation below spec.; Hf below spec.
	451	Strip	0.020" x 1.5" x 48"	0.9	NRC	2691	P.O. Chemistry, Tensile, Elongation, Grain size, Bend	---	No	Hf below spec.; C above spec.
509-(1-60)		Tube	0.375" OD x 0.008" wall	1.9	NRC	3352	SPPS-54-R1	Tensile Ultimate 80-111 ksi 0.2% Y.S. 65-100 ksi Elongation 20% min.	No	C above spec.; Hf below spec.

TABLE IV. (Cont'd)

Alloy	MCN Number	Mill Product		Weight	Vendor	Heat Number	Specifications			Meets All Specification Requirements	Remarks
		Form	Size				Number	Mech. Grade	Major Exceptions		
W-25Re	442	Wire	0.005" diameter x 2500'	--	Hoskins	Lot P985				---	---
	453	Wire	0.005" diameter x 2933'	--	Hoskins	Lot P986				---	---
W-3Re	443	Wire	0.005" diameter x 1650'	--	GE	2432		3D218CS		---	---
	464	Wire	0.005" diameter x 3950'	--	GE	Lot 27ABAA				---	---
464	464	Wire	0.005" diameter x 1970'	--	GE	2589A		3D218CLS		---	---
	464	Wire	0.005" diameter x 1970'	--	GE	Lot 3D31ADAB				---	---
465	465	Wire	0.005" diameter x 66'	--	GE	2589B		3D218CLS		---	---
	465	Wire	0.005" diameter x 66'	--	GE	Lot 3D31ACAB				---	---
T-222	489	Sheet	0.009" x 3.5" x 6.5"	--	Westinghouse	2360		3D218HF		---	---
	489	Sheet	0.009" x 3.5" x 6.5"	--	Westinghouse	Lot 3D26ADAA T-39-3				---	---

TABLE V. CHEMICAL ANALYSES OF REFRACTORY ALLOY MILL PRODUCTS

Alloy	MCN Number	Mill Product		Heat Number	Sample Source	Analyzed By	Chemical Analyses, ppm					
		Form	Size				C	O	N	H	Zr	
Cb-12r	400	Foill	0.002" x 3.5" x 125'	333-5	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	300	300	10	0.87/1.2	
401-(1-29)	402	Foill	0.002" x 0.5" x 10,800'	333-5	Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
481	482	Foill	0.002" x 3.5" x 125'	2590B	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
404-(1-12)	404-4	Tube	0.375" OD x 0.065" wall	11-229-01	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							30	90	45	7.8	1.1	
404-8	406	Tube	0.375" OD x 0.065" wall	11-229-01	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							30	90	45	7.8	1.1	
410-(1-204)	431-(1-6)	Tube	0.375" OD x 0.065" wall	355-70274	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
437-(1-22)	438	Tube	0.375" OD x 0.065" wall	98-70546	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
440-(1-2)	441-(1-3)	Tube	0.375" OD x 0.065" wall	98-70546	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
444	445-(1-2)	Tube	0.375" OD x 0.065" wall	98-70546	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
447	466-(1-55)	Tube	0.375" OD x 0.065" wall	11-052-1	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
467	470-(1-2)	Tube	0.375" OD x 0.065" wall	2590 F-4	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
405-(1-2)	411-(1-2)	Bar	2.5" diameter	FV-136	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	
412-(1-2)	412-(1-2)	Bar	3.25" diameter	FV-136	Ingot Final Product	Vendor	Max.	Max.	Max.	Max.	Max.	
							100	115	56	1	1	

TABLE V. (Cont'd)

MCN Number	Form	Mill Product	Size	Heat Number	Sample Source	Analyzed By	Chemical Analyses, ppm					
							C	O	N	H	2P, %	
413	Bar	3.5" diameter		FV-136	Final Product	Vendor	50	62	62	3	--	--
419	Bar	0.125" diameter x 72"		5120	Final Product	GE	40 ¹	62	34	3	--	--
					Ingot	Vendor	20 ¹	55 ¹	<10 ¹	1 ¹	0.99 ¹	
					Final Product	Vendor	70	136	31	2	--	
420-(1-4)	Bar	0.375" diameter		5155	Final Product	GE	95	188	56	3	--	
					Ingot	Vendor	49 ¹	21 ¹	19 ¹	9 ¹	1.1 ¹	
					Final Product	Vendor	10	14	22	2	--	
421-(1-2)	Bar	0.500" diameter		5155	Final Product	Vendor	20	8	12	3	--	
422-(1-9)	Bar	0.500" diameter		5120	Final Product	GE	30	109	48	6	--	
423-(1-6)	Bar	1.0" diameter		5155	Final Product	Vendor	10	115	42	2	--	
424-(1-6)	Bar	1.5" diameter		5155	Final Product	GE	40 ¹	18	21	1	--	
425-(1-5)	Bar	2.0" diameter		5154	Final Product	Vendor	10	1	10	5	--	
					Final Product	Vendor	3	2	14	5	--	
426-(1-20)	Bar	0.5" x 1.0"		5155	Ingot	Vendor	46 ¹	38	20 ¹	6 ¹	0.9	
					Final Product	Vendor	10	12	24	7	--	
427-(1-5)	Bar	1.0" x 1.0"		5155	Final Product	GE	25 ¹	22 ¹	22 ¹	9 ¹	--	
					Final Product	Vendor	10	<1	15	4	--	
428-(1-7)	Bar	1.25" diameter		5155	Final Product	GE	20 ¹	18	13	1	--	
					Final Product	Vendor	10	<1	10	4	--	
429-(1-3)	Wire	0.062" diameter		5154	Final Product	GE	15	15	13	1	--	
					Final Product	Vendor	10	6	23	7	--	
430	Wire	0.094" diameter		5154	Final Product	GE	30 ¹	18	41	2	--	
455	Wire	0.094" diameter		5154	Final Product	Vendor	40 ¹	59	25	1	--	
456	Wire	0.062" diameter		5154	Final Product	GE	56 ¹	57	11	5	--	
457	Wire	0.062" diameter		5123	Final Product	Vendor	20	32	31	5	--	
458	Wire	0.062" diameter		5155	Final Product	GE	2	15	41	2	--	
501	Wire	0.020" diameter		5155	Final Product	GE	2	33	100	8	--	
478	Rod	1.250" diameter x 250'		Ingot # 27605-D	Final Product	Vendor	5	16	420	0.7	--	
					Final Product	Vendor	50	75	30	2.1	1.1	
479	Rod	0.5" diameter x 24"		808849	Final Product	Vendor	70	70	25	2.6	--	
					Ingot	Vendor	25	90	40	<1	0.89	
493	Rod	0.375" diameter x 18"		808849	Final Product	Vendor	20	50	43	9	--	
					Final Product	Vendor	35 ¹	45	27	8	--	
494	Rod	0.5" diameter x 12"		808849	Ingot	Vendor	25	90	40	<1	0.89	
					Final Product	Vendor	10	30	50	3	--	
496	Rod	1.125" diameter x 6"		808971	Final Product	GE	110	68	40	9	--	
498	Bar	2" diameter x 6"		2701	Ingot	Vendor	22	86	56	1	1.12	
					Final Product	Vendor	30	79	49	46	--	
499	Bar	2.5" diameter x 18"		2701	Ingot	Vendor	22	66	56	1	1.12	
					Final Product	Vendor	22	66	56	1	1.12	
500	Bar	3.125" diameter x 17"		2701	Final Product	Vendor	20	50	60	1	--	
					Final Product	Vendor	70	55	48	9	--	
510	Rod	0.5" diameter x 48"		70559	Ingot	Vendor	<10	81	68	<5	0.90	
					Final Product	Vendor	<10	90	50	7.1	--	
407	Plate	0.250" x 12" x 50"		911-70341	Final Product	Vendor	101	97.5 ¹	61.5 ¹	9 ¹	--	
					Final Product	Vendor	110	141	48	8	--	
407	Plate	0.250" x 12" x 50"		911-70341	Ingot	Vendor	<10	81	68	<5	0.90	
					Final Product	Vendor	111	801	81	81	0.90	
407	Plate	0.250" x 12" x 50"		911-70341	Ingot	Vendor	<10	81	68	<5	0.90	
					Final Product	Vendor	<10	37	87	8.4	--	
407	Plate	0.250" x 12" x 50"		911-70341	Final Product	Vendor	121	471	70 ¹	9.5 ¹	--	
					Final Product	Vendor	301	1651	551	4.91	0.911	
407	Plate	0.250" x 12" x 50"		911-70341	Ingot	Vendor	<30	148	30	1.9	--	
					Final Product	Vendor	15	232	68	4	--	
407	Plate	0.250" x 12" x 50"		911-70341	Ingot	Vendor	60 ¹	90 ¹	45 ¹	4 ¹	0.971	
					Final Product	Vendor	<30	110	60	1	--	
407	Plate	0.250" x 12" x 50"		911-70341	Final Product	Vendor	401	84	44	<1	--	
					Final Product	Vendor	401	84	44	<1	--	

TABLE V. (Cont'd)

Alloy	MCN Number	Mill Product		Heat Number	Sample Source	Analyzed By	Chemical Analyses, ppm				
		Form	Size				C	O	N	H	Zr, %
Cb-12r	408-(1-3)	Plate	0.500" x 6" x 25"	912-1211	Ingot	Vendor	<30 ¹	145 ¹	65 ¹	5 ¹	1.04 ¹
					Final Product	Vendor	<30	80	50	4	--
		Plate	0.500" x 6" x 25"	911-70341	Final Product	GE	30 ¹	130	44	1	--
					Final Product	Vendor	<30	50	40	3	--
		Plate	0.500" x 6" x 9"	912-1018	Final Product	GE	45	83	31	1	--
					Ingot	Vendor	60 ¹	210 ¹	60 ¹	4 ¹	1.07 ¹
		Plate	0.500" x 6" x 25"	5194	Final Product	Vendor	40	200	65	4	--
					Final Product	GE	50 ¹	252	77	1	--
		Sheet	0.125" x 10" x 43"	912-1211	Ingot	Vendor	10	59	33	5	0.97
					Final Product	Vendor	60	190	135	4	--
	414	Sheet	0.0175" x 12" x 24"	912-1018	Final Product	GE	60	204	97	3	--
					Final Product	Vendor	<30	170	80	3	--
		Sheet	0.040" x 12" x 50"	912-70112	Final Product	GE	65	243	74	<1	--
					Ingot	Vendor	55 ¹	220 ¹	66 ¹	4 ¹	1.05 ¹
		Sheet	0.030" x 12" x 18"	911-70341	Final Product	Vendor	50	130	85	1	--
					Final Product	GE	35	142	22	2	--
		Sheet	0.032" x 10.5" x 60"	911-70341	Final Product	Vendor	30	90	30	<1	--
					Final Product	GE	60 ¹	125	48	2	--
		Sheet	0.040" x 12" x 50"	373-342	Final Product	Vendor	80	110	80	2	--
					Ingot	Vendor	35	60	39	5	0.89
D-43	432-(1-2)	Sheet	0.125" x 30" x 37"	912-1018	Final Product	Vendor	35	90	42	5	--
					Final Product	Vendor	40	90	50	2	--
		Sheet	0.032" x 10.5" x 60"	912-1189	Final Product	Vendor	35 ¹	180 ¹	87 ¹	4 ¹	1.0 ¹
					Final Product	Vendor	50	100	65	3	--
		Sheet	0.032" x 12.5" x 60"	912-1018	Final Product	Vendor	40	90	50	2	--
					Ingot	Vendor	--	--	--	--	1.08
		Sheet	0.0175" x 12" x 24"	808950	Final Product	Vendor	5	20	46	5	--
					Final Product	GE	50	281	91	1	--
	433	Tube	0.375" OD x 0.008" wall x 5'	43-473	Ingot	Vendor	Max. 800-1200	Max. 400	Max. 100	Max. 20	Zr 0.75-1.25% W 9-11%
					Final Product	Vendor	800	75	30	4	Zr 1.0 W 9.6
TZM	491-(1-35)	Tube	0.375" OD x 0.008" wall x 7"	43-473	Final Product	Vendor	870	42	45	10	--
					Final Product	GE	830	133	37	11	2x0.096 Ti 0.50
		Tube	0.375" OD x 0.008" wall x 7"	43-473	Final Product	Vendor	880 ¹	159	46	<1	--
					Final Product	GE	200	<2	1	<1	Zr 0.095 Ti 0.48
	435-(1-2)	Bar	1.0" diameter	7474	Final Product	Vendor	Max. 100-400	Max. 25	Max. 5	Max. 1	Zr 0.06-0.12% Ti 0.4-0.35%
					Final Product	Vendor	210	<7	<8	<1	Zr 0.079 Ti 41
		Bar	0.875" diameter	7473	Final Product	Vendor	175	19	7	1	--
					Final Product	GE	160	2	<1	<1	2x0.096 Ti 0.50
		Bar	2.0" diameter	7501	Final Product	Vendor	145	8	4	<1	--
					Final Product	GE	200	<2	1	<1	Zr 0.095 Ti 0.48
	461	Bar	1.0" diameter	7479	Final Product	Vendor	155	22	14	1	--
					Final Product	GE	140	<4	2	<1	Zr 0.086 Ti 0.51
		Bar	0.5" diameter	7436	Final Product	Vendor	140 ¹	17	6	1	--
					Final Product	GE	150	<5	1	<1	Zr 0.083 Ti 0.52
		Bar	2" diameter	7555	Final Product	Vendor	150	<5	<1	<1	Zr 0.096 Ti 0.55
					Final Product	Vendor	210	<4	<1	<1	Zr 0.103 Ti 0.49

TABLE V. (Cont'd)

Alloy	MCN Number	Mill Product		Heat Number	Sample Source	Analyzed By	Chemical Analyses, Ppm					
		Form	Size				C	O	N	H	Other	
Ti	460	Foil	0.003" x 6"	60165-Ti-53B	Ingot	SPECIFICATION	Max. 300	Max. 300	Max. 150	Max. 15		
	477	Foil	0.002" x 8" x 620"	60165-Ti-53B	Ingot	Vendor	401	<50 ¹	201	31		
						Vendor	Top 50	--	--	3.3		
						GE	Bottom <30	<50	20	3.4	<50 Zr	
	480	Foil	0.002" x 8" x 315"	61029N-Ti	Final Product	Vendor	50	--	20	4		
					Final Product	Vendor	Top	--	--	3.8		
					Final Product	GE	Bottom <30	<50	20	4.1	<100 Zr	
					Final Product	GE	Top	251	37	9	4	
	471	Sheet	0.062" x 2.125" x 32"	TM 133	Final Product	Vendor	11	60	3	5		
	472	Sheet	0.032" x 0.750" x 12"	EB-80	Final Product	Vendor	80	90	3	6		
T-111	474	Rod	0.250" diameter x 24"	81159	Final Product	Vendor	<10	9	90	3		
	475	Rod	0.625" diameter x 12"	81167	Final Product	Vendor	30	8	12	6		
	476	Bar	1.125" diameter x 8"	81166	Final Product	Vendor	70	7	12	7		
	483	Wire	0.020" diameter x 7200"	81159	Final Product	Vendor	<10	9	90	3		
					Final Product	GE	50-80	145	18	1		
					Final Product	GE	Max. 30	Max. 100	Max. 70	Max. 15	W 7-9 HF 2-2.8	
	448	Bar	0.1875" diameter x 12"	2650	Ingot	Vendor	291	101	271	--	W 7.31 HF 1.81	
					Final Product	Vendor	30	11	65	42	W 7.1 HF 1.8	
	449	Bar	1.0" diameter x 12"	3171	Final Product	GE	201	66	22	1	W 8.0 HF 1.01	
					Ingot	Vendor	23	28	31	--	W 8.3 HF 2.1	
W-25Zr					Final Product	Vendor	26	17	23	20	W 7.11 HF 1.71	
	450	Strip	0.015" x 1.5" x 48"	2691	Final Product	GE	401	26	24	4	W 7.11 HF 1.71	
					Ingot	Vendor	191	<101	261	--	W 7.11 HF 1.71	
					Final Product	Vendor	53	324	39	42	W 7.11 HF 1.8	
	451	Strip	0.020" x 1.5" x 48"	2691	Final Product	GE	201	37	38	1	--	
					Final Product	Vendor	54	84	40	<2	W 7.3 HF 1.8	
					Final Product	Vendor	Max. 75	Max. 300	Max. 100	Max. 10	W 7-9 HF 1.8-2.4	
	509-(1-50)	Tube	0.375" OD x 0.008" wall	3352	Ingot	SPECIFICATION	63	19	26	--	W 8.0 HF 2.1	
					Final Product	Vendor	241	170	39	2	--	
					Final Product	Vendor	1431	--	--	--	--	
W-25Zr	442	Wire	0.005" diameter x 2500'	PB85	Final Product	Vendor	--	--	--	--	Re-26.26	
	453	Wire	0.005" diameter x 2933'	PB86	Final Product	Vendor	--	--	--	--	Re 25.5	
	489	Sheet	0.009" x 3.5" x 6.5"	Ta-39-3	Final Product	Vendor	130	18	41	--	W 8.9 HF 2.2	
					Final Product	GE	290	162	28	<1		

1 Average of Two Analyses.

2 Average of Four Analyses.

TABLE VI. MECHANICAL PROPERTIES AND GRAIN SIZE OF REFRACTORY ALLOY MILL PRODUCTS

[illegible]

TABLE VI.

Alloy	MCN Number	Mill Products		Heat Number	Room Temperature Tensile Properties			2000°F Stress-Rupture Life at 10,000 psi			Hardness			Grain Size				
		Form	Size		Final Heat Treatment	Ult. Ksi	0.2% Y.S. Elong.		Hours	Rupture Life, %	Bulk	Surface		Trans. ASTM Number	Maximum Longitudinal Length, Inch			
							Ksi	%				Flare	Micro Center					
Cb-12r	422-(1-9)	Bar	0.500" diameter	5120	48.0 ¹	27.9 ¹	41 ¹	2200°F/1 hour	>30 ¹	0.7	---	---	101DPH	7	---			
	423-(1-6)	Bar	1.0" diameter	5155	-	-	-	2200°F/1 hour	---	---	---	---	---	(6 GE)	---			
	424-(1-6)	Bar	1.5" diameter	5155	36.6 ¹	24.1 ¹	44 ¹	2200°F/1 hour	>30 ¹	26 ¹	---	---	100DPH	6-7	---			
	425-(1-5)	Bar	2.0" diameter	5154	partially recrystallized	39.4 ¹	34.8 ¹	37 ¹	15	41	---	---	---	108DPH	6	---		
	426-(1-20)	Bar	0.5" x 1.0"	5155	38.9 ¹	14.7 ¹	43 ¹	2200°F/1 hour	29	45	---	---	---	112DPH	5-6	---		
	427-(1-5)	Bar	1.0" x 1.0"	5155	-	-	-	2200°F/1 hour	24	55	---	---	---	97DPH	5-6	< 0.010		
	428-(1-7)	Bar	1.25" diameter	5155	-	-	-	2200°F/1 hour	---	---	---	---	---	97DPH	5-7	0.010		
	429-(1-3)	Wire	0.062" diameter	5154	-	-	-	2200°F/1 hour	---	---	---	---	---	97DPH	5-6	---		
	430	Wire	0.094" diameter	5154	-	-	-	2200°F/1 hour	---	---	---	---	---	---	---	6-7	---	
	478	Bar	1.250" diameter x 24"	808849	35.2 ¹	18.8 ¹	38.5 ¹	2200°F/1 hour	---	---	---	---	---	---	---	6-7	---	
	479	Bar	0.5" diameter x 24"	808849	40.1 ¹	20.9 ¹	41 ¹	2200°F/1 hour	---	---	---	---	---	---	---	3-6	0.05	
	493	Bar	0.375" diameter x 18"	808791	38.9 ¹	23.4 ¹	40 ¹	2200°F/1 hour	---	---	---	---	---	---	---	5	< 0.010	
	494	Bar	0.5" diameter x 12"	808971	-	-	-	2200°F/1 hour	---	---	---	---	---	---	---	6	< 0.050	
	495	Bar	0.750" diameter x 12"	808971	-	-	-	2200°F/1 hour	---	---	---	---	---	---	---	5-1/2 - 6	< 0.050	
	496	Bar	1.125" diameter x 6"	808971	42.4 ¹	25.2 ¹	34.5 ¹	2200°F/1 hour	---	---	---	---	---	---	---	6	< 0.050	
	498	Bar	2" diameter x 6"	2701	partially recrystallized	36.9 ¹	22.5 ¹	29 ¹	>29	-	---	---	---	---	---	5 - 5-1/2	< 0.050	
	499	Bar	2.5" diameter x 18"	2701	partially recrystallized	36.8 ¹	25.3 ¹	25.5 ¹	>31.5 ¹	-	---	---	---	---	---	--	---	
	500	Bar	3.125" diameter x 17"	2701	recrystallized	38.8 ¹	26.6 ¹	23	2400°F/1 hour; partially recrystallized	36	-	---	---	---	---	--	---	
	510	Bar	0.5" diameter x 48"	70559	42 ¹	26.2 ¹	41 ¹	-	2200°F/1 hour	---	-	---	---	---	---	--	---	
	407	Plate	0.250" x 12" x 50"	911-70341	partially recrystallized	39.6 ¹	27.2 ¹	44 ¹	2200°F/1 hour; partially recrystallized	---	-	---	---	---	---	7	0.004	
	408-(1-3)	Plate	0.500" x 6" x 25"	912-1211	partially recrystallized	43.4 ¹	26.7 ¹	29 ¹	2200°F/1 hour; partially recrystallized	>30	-	---	---	---	---	8.5	< 0.020	
	414	Plate	0.500" x 6" x 25"	911-70341	partially recrystallized	37.3 ¹	24.2 ¹	49 ¹	2200°F/1 hour; partially recrystallized	>30	2.5	---	---	---	---	6	< 0.010	
	416-(1-2)	Plate	0.500" x 6" x 9"	912-1018	partially recrystallized	42.9 ¹	31.9 ¹	33 ¹	2200°F/1 hour; partially recrystallized	>30	0.5	---	---	---	---	6	< 0.020	
	468-(1-2)	Plate	0.250" x 5" x 72"	5194	54.3 ¹	38.5 ¹	22 ¹	>96	2200°F/1 hour	13.7	-	---	---	---	---	--	---	
	409-(1-4)	Sheet	0.0175" x 12" x 24"	912-1211	partially recrystallized	48.4 ¹	36.4 ¹	17 ¹	2200°F/1 hour; partially recrystallized	---	-	---	---	---	---	9.5	---	
	415-(1-4)	Sheet	0.125" x 10" x 43"	912-1018	partially recrystallized	43.8 ¹	31.7 ¹	30 ¹	2200°F/1 hour; partially recrystallized	---	-	---	---	---	---	7.5	< 0.010	
	417	Sheet	0.040" x 12" x 50"	912-70112	partially recrystallized	39.5 ¹	22.1 ¹	29 ¹	2200°F/1 hour; partially recrystallized	---	-	---	---	---	---	7.5	< 0.010	
	418-(1-4)	Sheet	0.030" x 12" x 18"	911-70341	partially recrystallized	46.2 ¹	34.5 ¹	20 ¹	2200°F/1 hour; partially recrystallized	---	-	---	---	---	---	7.5	< 0.005	
	432-(1-2)	Sheet	0.125" x 30" x 37"	911-70341	2200°F/1 hour	44.9 ¹	33.3 ¹	29 ¹	2200°F/1 hour	---	-	---	---	---	---	7	0.032	
	454	Sheet	0.040" x 12" x 50"	373-3A2	-	-	-	-	2200°F/1 hour	---	-	---	---	---	---	--	---	
	459	Sheet	0.032" x 10.5" x 60"	912-1018	-	-	-	-	2200°F/1 hour	---	-	---	---	---	---	108VBN	7.5	0.0088
	462-(1-2)	Sheet	0.032" x 12.5" x 60"	912-1189	-	-	-	-	2200°F/1 hour	---	-	---	---	---	---	107VBN	7.5	0.0144

TABLE VI. (Cont'd)

Alloy	MCN Number	Mill Product Form	Size	Heat Number	Final Heat Treatment	Room Temperature Tensile Properties		2000°F Stress Rupture Life at 10,000 psi	Hardness		Grain Size			
						Ult. Ksi	0.2% Y.S. Ksi		Elong. %	Flare	Bulk	Surface	Micro Center	Trans. ASTM Number
Cb-1Zr	463	Sheet	0.032" x 12.5" x 60"	912-1018	2200°F/1 hour;	-	--	--	----	--	--	108VHN	7.5	0.0088
	497	Sheet	0.0175" x 12" x 24"	808980	2200°F/1 hour	42.9 ¹	29.9 ¹	25.5 ¹	----	--	--	49.5R ₃₀ T	6.5	< 0.050
D-43	490	Tube	0.375" OD x 0.008" wall x 5'	43-473	Annealed 2850°F/1 hour	74.7	49.4	23.5	--	--	--	140DPH	7-8	---
	491-(1-35)	Tube	0.375" OD x 0.008" wall x 7"	43-473	Annealed 2850°F/1 hour	74.7	49.4	23.5	--	--	--	140DPH	7-8	---
TZM	492-(1-7)	Tube	0.375" OD x 0.008" wall x 7"	43-473	Annealed 2850°F/1 hour	74.7	49.4	23.5	--	--	--	140DPH	7-8	---
	433	Bar	0.125" diameter	4-3934	Stress-relieved 2200°F/1/4 hour	-	--	--	----	--	--	260-274DPH	7-8	---
	434	Bar	0.875" diameter	7473	Stress-relieved 2300°F/3/4 hour	125.2 ¹	107.8 ¹	29 ¹	----	--	--	276-287DPH	-	---
	435-(1-2)	Bar	1.0" diameter	7474	Stress-relieved 2300°F/3/4 hour	122.1 ¹	103.3 ¹	28 ¹	----	--	--	268-276DPH	-	---
	436	Bar	2.0" diameter	7501	Room Temperature 2000°F (GE)	104.0	102.3	30	----	--	--	--	-	---
	461	Bar	1.0" diameter	7479	Stress-relieved 2300°F/1 hour	65.9	65.0	14	----	--	--	268-274DPH	-	---
	469	Bar	0.5" diameter	7436	Stress-relieved 2350°F/3/4 hour	121.7 ¹	103.8 ¹	23 ¹	----	--	--	272-281DPH	-	---
	473	Bar	2" diameter	7555	Stress-relieved 2200°F/1/2 hour	131 ¹	122.8 ¹	29 ¹	----	--	--	276-306DPH	-	---
Ta	477	Foil	0.002" x 8" x 620"	60165-Ta-53B	Stress-relieved 2350°F/1 hour	104.9 ¹	89.1 ¹	17 ¹	----	--	--	268-272DPH	-	---
	480	Foil	0.002" x 8" x 315"	60219N-Ta	Unannealed	-	--	--	----	--	--	62BHN	-	---
	472	Sheet	0.032" x 0.750" x 12"	EB-80	Fully annealed	-	--	--	----	--	--	70BHN	-	---
	474	Bar	0.250" diameter x 24"	81159	Fully annealed	-	--	--	----	--	--	--	-	---
	475	Bar	0.625" diameter x 12"	81167	Fully annealed	-	--	--	----	--	--	--	-	---
	476	Bar	1.125" diameter x 8"	81166	Fully annealed	-	--	--	----	--	--	--	-	---
T-111	483	Wire	0.020" diameter x 7200"	81159	Unannealed	-	--	--	----	--	--	--	-	---
	448	Bar	0.1875" diameter x 12"	2650	Annealed 2800°F/1.5 hours	85.9	76.4	25	----	--	--	50Ra	8-10	---
	449	Bar	1.0" diameter x 12"	3171	Annealed 2800°F/1.5 hours	93.2	81.2	26	----	--	--	50Ra	8-10	---
	450	Strip	0.015" x 1.5" x 48"	2691	Annealed 2800°F/1.5 hours	87.5	83.6	10	----	--	--	27Ra	7-9	---
	451	Strip	0.020" x 1.5" x 48"	2691	Annealed 2800°F/1.5 hours	81.0	70.4	21.0	----	--	--	31Ra	8-10	---
						86.6 ¹	77.9 ¹	16 ¹ (GE)						
						53 ¹	42.8 ¹	13 ¹ (800°F air)						
						60 ¹	44.4 ¹	19 ¹ (800°F vacuum)						
						57.5 ¹	42.8 ¹	13 ¹ (1000°F vacuum)						
						80-111	65-100	20 Min.						
								2400°F						
								19,000 psi						
SPECIFICATION														ASTM 6
509-(1-50)	Tube	0.375" OD x 0.008" wall	3352	Annealed 2700°F/1.5 hours	98.2	78.1	28	30	----	--	--	250 Knoop	7.5	---

TABLE VI. (Cont'd)

Alloy	MCN Number	Mill Product		Heat Number	Final Heat Treatment	Room Temperature Tensile Properties			2000°F Stress Rupture Life		Hardness		Grain Size	
		Form	Size			Ult. Ksi	0.2% Y.S. Ksi	Elong. %	Hours	at 10,000 psi	Bulk	Surface	Trans. ASTM Number	Maximum Longitudinal Length, Inch
W-25Re 442	442	Wire	0.005" diameter x 2500'	PB855	Annealed, partially recrystallized	-	-	-	-	-	-	-	-	-
		Wire	0.005" diameter x 2833'	PB85	Annealed, partially recrystallized	-	-	-	-	-	-	-	-	-
W-3Re 443	443	Wire	0.005" diameter x 1850'	2432	Annealed	88.0	-	-	-	-	-	-	-	-
		Wire	0.005" diameter x 3950'	2589A	1750°C	50.1	-	1.4	-	-	-	-	-	-
465	465	Wire	0.005" diameter	2549B	1750°C	49.2	-	2.4	-	-	-	-	-	-
		Wire	0.020" x 66'	2360	>1750°C	38.6	-	20.4	-	-	-	-	-	-

¹ Average of Two Analyses.

TABLE VII. RESULTS OF NONDESTRUCTIVE QUALITY ASSURANCE TESTS OF REFRACTORY ALLOY MILL PRODUCTS

Alloy	MCN Number	Mill Product		Heat Number	Nondestructive Tests		
		Form	Size		Penetrant	Ultrasonic	Hydrostatic
Cb-12r	400	Foil	0.002" x 3.5" x 125'	333-5	Surface covered with oil	-----	-----
	401-(1-29)	Foil	0.002" x 0.5" x 10,800'	333-5	Surface covered with oil	-----	-----
	402	Foil	0.005" x 3.0" x 8'	333-5	-----	-----	-----
	481	Foil	0.002" x 3.5" x 125'	2590B	-----	-----	-----
	482	Foil	0.002" x 0.5" x 1350'	2590B	-----	-----	-----
	404-(1-12)	Tube	0.375" OD x 0.65" wall	11-229-01	Not performed; ID surface rough	1-4 tubes rejected for cracks & internal defects < 3% wall thickness	-----
	406	Tube	2" OD x 0.25" wall x 4'	11-229-02	-----	-----	-----
	410-(1-204)	Blank					
		Tube	0.375" OD x 0.008" wall x 7"	355-70274	Passed; ID 7/13 rms; OD 24/30 rms	-----	Modified test passed
	431-(1-6)	Tube	0.1875" OD x 0.025" wall	355-70303	100% passed	100% passed	100% passed; 3500 psi, 1 minute
	437-(1-22)	Tube	0.375" OD x 0.065" wall	98-70546	100% passed	100% passed	100% passed; 4150 psi, 5 seconds
	438	Tube	0.275" OD x 0.125" wall	5-53003	100% passed	100% passed	-----
	439	Tube	3.25" OD x 0.125" wall	5-53003	100% passed	100% passed	-----
	440-(1-2)	Tube	0.375" OD x 0.065" wall	98-70546	-----	100% passed; one major defect in each tube (removed)	-----
	441-(1-3)	Tube	1.0" OD x 0.100" wall	98-70546	100% passed	100% passed; one major defect in each tube; N12"	100% passed; 2400 psi, 5 seconds
	444	Tube	0.25" OD x 0.062" wall	98-70546	100% passed	100% passed	100% passed; 5952 psi, 5 seconds
	445-(1-2)	Tube	0.375" OD x 0.065" wall	98-70546	100% passed; 445-1 micro shows rejectable defect	100% passed	100% passed; 4160 psi, 5 seconds
	447	Tube	0.375" OD x 0.065" wall	98-70546	100% passed	100% passed	100% passed; 4160 psi, 5 seconds
	466-(1-55)	Tube	0.375" OD x 0.008" wall	11-052-1	-----	-----	-----
	467	Tube	0.375" OD x 0.008" wall x 5'	11-052-1	-----	-----	-----
	470-(1-2)	Tube	0.675" OD x 0.125" wall x 78"	2590F-4	-----	100% passed; 470-1 has rejectable indications	100% passed; 4500 psi
	405-(1-2)	Bar	2.5" diameter	FV-136	100% passed	100% passed	-----
	411-(1-2)	Bar	3.0" diameter	FV-136	100% passed	100% passed	-----
	412-(1-2)	Bar	3.25" diameter	FV-136	100% passed	100% passed	-----
	413	Bar	3.5" diameter	FV-136	100% passed	100% passed	-----
	419	Bar	0.125" diameter x 72"	5120	-----	100% passed	-----
	420-(1-4)	Bar	0.375" diameter	5155	100% passed	100% passed	-----

TABLE VII. (Cont'd)

Alloy	MCN Number	Mill Product		Heat Number	Nondestructive Test	
		Form	Size		Penetrant	Ultrasonic
Cb-1Zr	421-(1-2)	Bar	0.500" diameter	5155	100% passed	100% passed
	422-(1-9)	Bar	0.500" diameter	5120	100% passed; small tears	100% passed
	423-(1-6)	Bar	1.0" diameter	5155	100% passed	100% passed
	424-(1-6)	Bar	1.5" diameter	5155	100% passed	100% passed
	425-(1-5)	Bar	2.0" diameter	5154	100% passed	100% passed
	426-(1-20)	Bar	0.5" x 1.0"	5155	100% passed	100% passed
	427-(1-5)	Bar	1.0" x 1.0"	5155	100% passed	100% passed
	428-(1-7)	Bar	1.25" diameter	5155	100% passed	100% passed
	429-(1-3)	Wire	0.062" diameter	5154	100% passed	100% passed
	430	Wire	0.094" diameter	5154	-----	-----
	455	Wire	0.094" diameter	5154	-----	-----
	456	Wire	0.062" diameter	5154	-----	-----
	457	Wire	0.062" diameter	5123	-----	-----
	458	Wire	0.062" diameter	5155	-----	-----
	478	Rod	1.250" diameter x 24"	80B849	100% passed	100% passed
	479	Rod	0.5" diameter x 24"	80B849	100% passed	100% passed
	493	Rod	0.375" diameter x 18"	80B971	100% passed	100% passed
	494	Rod	0.5" diameter x 12"	80B971	100% passed	100% passed
	495	Rod	0.750" diameter x 12"	80B971	100% passed	100% passed
	496	Rod	1.125" diameter x 6"	80B971	100% passed	100% passed
	498	Bar	2" diameter x 6"	2701	100% Passed	100% Passed
	499	Bar	2.5" diameter x 18"	2701	100% passed	100% passed
	500	Bar	3.125" diameter x 17"	2701	100% passed	100% passed
	510	Rod	0.5" diameter x 48"	70559	100% passed	100% passed
	407	Plate	0.250" x 12" x 50"	911-70341	100% passed	100% passed
	408-(1-3)	Plate	0.500" x 6" x 25"	912-1211	100% passed	100% passed
	414	Plate	0.500" x 6" x 25"	911-70341	100% passed	100% passed
	416-(1-2)	Plate	0.500" x 6" x 9"	912-1018	100% passed	100% passed
	468-(1-2)	Plate	0.250" x 5" x 72"	5194	100% passed	100% passed
	409-(1-4)	Sheet	0.0175" x 12" x 24"	912-1211	100% passed	100% passed
	415-(1-4)	Sheet	0.125" x 10" x 43"	912-1018	100% passed	100% passed
	417	Sheet	0.040" x 12" x 50"	912-70112	100% passed	100% passed
	418	Sheet	0.030" x 12" x 18"	911-70341	100% passed	100% passed
	432-(1-2)	Sheet	0.125" x 30" x 37"	911-70341	100% passed	100% passed
	454	Sheet	0.040" x 12" x 50"	373-3A2	-----	-----
	459	Sheet	0.032" x 10.5" x 60"	912-1018	-----	-----
	462-(1-2)	Sheet	0.032" x 12.5" x 60"	912-1189	-----	-----
	463	Sheet	0.032" x 12.5" x 60"	912-1018	-----	-----

TABLE VII. (Cont'd)

Alloy	MCN Number	Form	Size	Heat Number	Nondestructive Tests	
					Penetrant	Ultrasonic
Cb-12r	497	Sheet	0.0175" x 12" x 24"	80B950	100% passed	100% passed
	490	Tube	0.375" OD x 0.008" wall x 5'	43-473	OD & ID surface unacceptable, 0.001" cracks	100% passed
TZM	491	Tube	0.375" OD x 0.008" wall x 7"	43-473	-----	100% passed
	492-(1-7)	Tube	0.375" OD x 0.008" wall x 7"	43-473	-----	100% passed
	433	Bar	0.125" diameter	4-3934	100% passed	100% passed
	434	Bar	0.875" diameter	7473	100% passed	100% passed
	435-(1-2)	Bar	1.0" diameter	7474	100% passed	100% passed
	436	Bar	2.0" diameter	7501	100% passed	100% passed
	461	Bar	1.0" diameter	7479	100% passed	-----
	469	Bar	0.5" diameter	7436	100% passed	-----
T-111	473	Bar	2.0" diameter	7555	100% passed	100% passed
	448	Bar	0.1875" diameter x 12"	2650	-----	-----
	449	Bar	1.0" diameter x 12"	3171	-----	16 defects greater than accepted limits
	450	Strip	0.015" x 1.5" x 48"	2691	-----	100% passed
	451	Strip	0.020" x 1.5" x 48"	2691	-----	100% passed
	509-(1-50)	Tube	0.375" OD x 0.008" wall	3352	100% passed	-----

TABLE VIII. SUMMARY OF OVERALL QUALITY ASSURANCE TEST RESULTS

Material	Form	Number of Lots	Chemistry		Tensile Properties		Stress-Rupture		Hardness		Grain Size		Penetrant*		Ultrasonic*		Hydrostatic*		Flare	
			Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed
Cb-lZr	Foil	5	3	2	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Cb-lZr	Tube	15	14	1	14	0	5	0	11	0	10	2	241	0	98	10	240	0	10	0
Cb-lZr	Bar	24	24	0	16	0	10	1	23	0	20	0	81	0	82	0	--	--	--	--
Cb-lZr	Wire	7	7	0	--	--	--	--	--	--	2	0	--	--	--	--	--	--	--	--
Cb-lZr	Plate	5	5	0	5	0	4	0	5	0	4	0	9	0	9	0	--	--	--	--
Cb-lZr	Sheet	10	10	0	6	0	--	--	10	0	9	0	16	0	16	0	--	--	--	--
D-43	Tube	2	2	0	--	--	--	--	--	--	2	0	--	--	2	0	--	--	2	0
TZM	Bar	7	7	0	6	0	--	--	7	0	--	--	8	0	6	0	--	--	--	--
Ta	Foil	3	3	0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Ta	Sheet	2	2	0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Ta	Bar	3	3	0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Ta	Wire	1	1	0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
T-111	Bar	2	0	2	2	0	--	--	--	--	2	0	--	--	0	1	--	--	--	--
T-111	Strip	2	0	2	1	1	--	--	--	--	2	0	--	--	2	0	--	--	--	--
T-111	Tube	1	0	1	1	0	1	0	--	--	1	0	1	0	--	--	--	--	1	0

* Total Number of Pieces Tested.

TABLE IX. SUMMARY OF INTERSTITIAL ELEMENT CONTENT OF Cb-1Zr ALLOY MILL PRODUCTS

Section Size, Inch	Mill Shape	Vendor	Ingot	Oxygen Analyses, ppm		
				Final Product		
				No. of Analyses	Range	Average
0.002 - 0.005	Foil	Kawecki	126	5	82-338	160
0.008	Tube	Superior	250	2	660-868	764
0.0175	Sheet	Wah Chang	145	2	190-204	197
0.025	Tube	Wah Chang	260	2	205-290	248
0.030-0.040	Sheet	Wah Chang	152	8	60-142	103
0.065	Tube	Wah Chang	162	12	110-303	154
0.062-0.094	Wire	Wah Chang	38	3	32-59	49
0.100	Tube	Wah Chang	200	2	200-226	213
0.125	Tube	Wah Chang	210	2	200-230	215
0.125	Rod	Union Carbide	55	2	156-188	172
0.375	Rod	Union Carbide	21	1	14	14
0.500	Rod	Union Carbide	38	4	8-115	63
1.0	Rod	Union Carbide	21	1	1	1

TABLE IX. (Cont'd)

Nitrogen Analyses, ppm						
Section Size, Inch	Mill Shape	Vendor	Ingot	Final Product		
				No. of Analyses	Range	Average
0.002 - 0.005	Foil	Kawecki	38	5	29-102	55
0.008	Tube	Superior	65	2	72-106	89
0.0175	Sheet	Wah Chang	65	2	97-135	116
0.025	Tube	Wah Chang	55	2	65-81	73
0.030 - 0.040	Sheet	Wah Chang	59	7	22-85	49
0.065	Tube	Wah Chang	35	12	<1-80	52
0.062 - 0.094	Wire	Wah Chang	20	3	11-31	22
0.100	Tube	Wah Chang	55	2	35-64	50
0.125	Tube	Wah Chang	38	2	20-70	45
0.125	Rod	Union Carbide	<10	2	31-56	44
0.375	Rod	Union Carbide	19	1	22	22
0.500	Rod	Union Carbide	15	4	12-48	31
1.0	Rod	Union Carbide	19	1	10	10

TABLE IX. (Cont'd)

Section Size, Inch	Mill Shape	Vendor	Ingot	Hydrogen Analyses, ppm		
				No. of Analyses	Range	Average
0.002 - 0.005	Foil	Kawecki	4	5	1-12	5
0.008	Tube	Superior	3	2	9-10	10
0.0175	Sheet	Wah Chang	5	2	3-4	4
0.025	Tube	Wah Chang	4	2	3-3	3
0.030 - 0.040	Sheet	Wah Chang	4	7	1-5	2
0.065	Tube	Wah Chang	5	12	1-9	4
0.062 - 0.094	Wire	Wah Chang	6	3	1-5	4
0.100	Tube	Wah Chang	3	2	2-6	4
0.125	Tube	Wah Chang	3	2	3-4	4
0.125	Rod	Union Carbide	1	2	2-3	3
0.375	Rod	Union Carbide	9	1	2	2
0.500	Rod	Union Carbide	5	4	1-6	3
1.0	Rod	Union Carbide	9	1	5	5

TABLE IX. (Cont'd)

Section Size, Inch	Mill Shape	Vendor	Ingot	Carbon Analyses, ppm		
				No. of Analyses	Range	Average
0.002 - 0.005	Foil	Kawecki	50	5	30-122	77
0.008	Tube	Superior	90	2	195-210	203
0.0175	Sheet	Wah Chang	<30	2	60-60	60
0.025	Tube	Wah Chang	40	2	90-120	105
0.030 - 0.040	Sheet	Wah Chang	49	7	30-60	43
0.065	Tube	Wah Chang	56	12	20-80	50
0.062 - 0.094	Wire	Wah Chang	46	3	20-56	39
0.100	Tube	Wah Chang	65	2	40-90	65
0.125	Tube	Wah Chang	55	2	30-30	30
0.125	Rod	Union Carbide	20	2	70-95	83
0.375	Rod	Union Carbide	49	1	10	10
0.500	Rod	Union Carbide	35	4	10-40	25
1.0	Rod	Union Carbide	49	1	10	10

TABLE X. PROCESS SPECIFICATIONS

<u>Title</u>	<u>Revised Specification No.</u>	<u>Original Specification No.</u>
1. Welding of Columbium-1% Zirconium Alloy by the Inert Gas Tungsten Arc Process	03-0004-00-B	SPPS-3B
2. Welding of Columbium-1% Zirconium Alloy by the Inert Gas Tungsten Arc Process	03-0005-00-A	SPPS-3C
3. Vacuum Brazing Bimetallic Tube Joints	03-0008-00-B	SPPS-9A
4. Operator Certification for High Temperature Vacuum Furnace Brazing of Bimetallic Tube Joints	03-0009-00-A	SPPS-10
5. Chemical Cleaning of Columbium and Columbium Alloy Products	03-0010-00-B	SPPS-11A
6. Grit Blasting Columbium and Columbium Alloy Products	03-0011-00-A	SPPS-12
7. Welding Columbium-1% Zirconium Alloy by the Electron Beam Process	03-0012-00-A	SPPS-14
8. Mass Spectrometric Leak Detection Using Helium	03-0013-00-B	SPPS-26
9. Welding of Austenitic Stainless Steel	03-0014-00-B ¹	SPPS-41
10. Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium, and Tantalum Alloys	03-0015-00-A	SPPS-50
11. Stainless Steel Conoseal Tube Unions	03-0016-00-A	SPPS-53

(1) Specification was Revised to Incorporate Changes in Radiographic Inspection Requirements Subsequent to its Use in Contract NAS 3-2547.

TABLE XI. ALKALI METAL PROCUREMENT AND PURIFICATION SPECIFICATIONS

<u>Title</u>	<u>Revised Specification No.</u>	<u>Original Specification No.</u>
1. Reactor Grade Sodium Metal	01-0031-00-B ¹	SPPS-45-I
2. Hot Trapped Reactor Grade Sodium Metal	01-0032-00-B ²	SPPS-45-II
3. High Purity Grade Potassium Metal	01-0033-00-B ¹	SPPS-46-I
4. Hot Trapped High Purity Grade Potassium Metal	01-0034-00-B ²	SPPS-46-II
5. Alkali Metal Handling and Control Procedures	03-0018-00-A	---

¹ Specifications were Revised to Incorporate Changes in the Maximum Allowable Impurities Subsequent to Their Use in Contract NAS 3-2547.

² Specifications were Revised to Incorporate Changes in the Maximum Allowable Impurities and in the Length of Time Required for Hot Trapping Subsequent to Their Use in Contract NAS 3-2547.

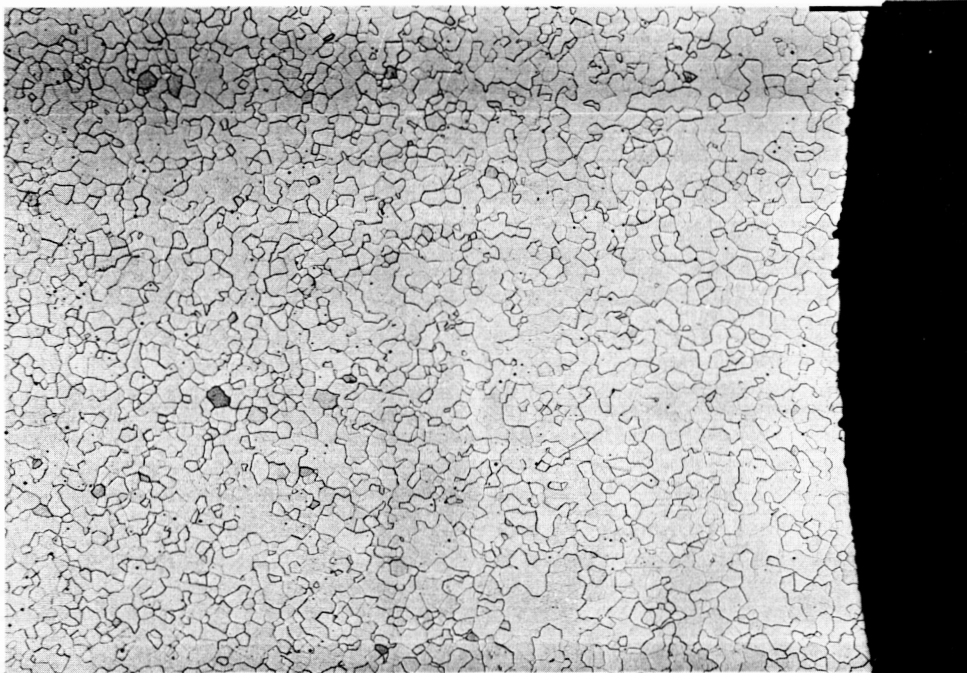


Figure 1. Microstructure of Transverse Section of Cb-1Zr Alloy 0.375-Inch OD x 0.065-Inch Thick Wall Tube (MCN 404-4) Used in the Construction of the Prototype Loop. Final Heat Treatment was One Hour at 2200°F.
(Z4265)
Etchant: 20%HF-20%HNO₃-60%Water Mag: 100X

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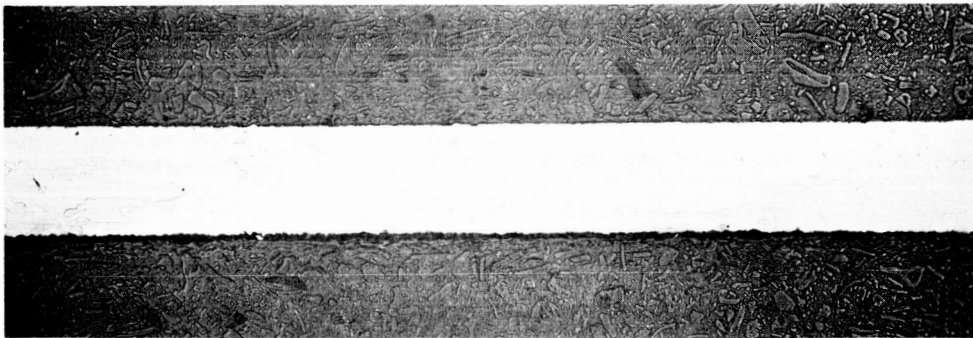
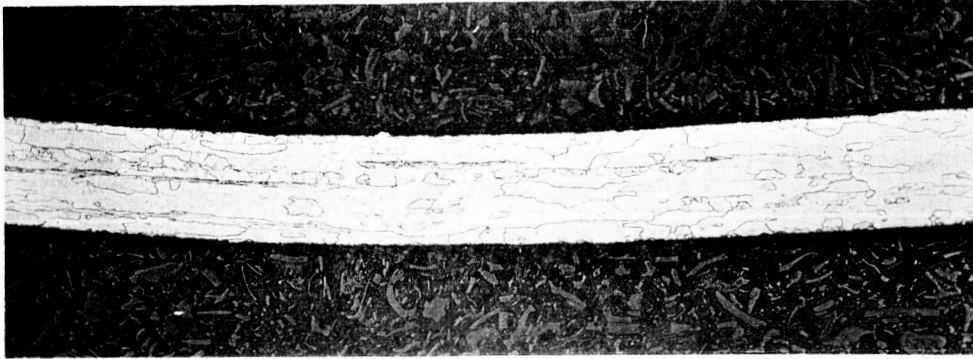


Figure 2. Microstructure of Cb-1Zr Alloy 0.375-Inch OD x 0.008-Inch Thick Wall Tube (MCN 410) Used for the Fabrication of Bellows for the Prototype Loop. Final Heat Treatment was One Hour at 2300°F. Top: Transverse Section (K211); Bottom: Longitudinal Section (K65).

Etchant: 20%HF-20%HNO₃-60%Water

Mag: 100X

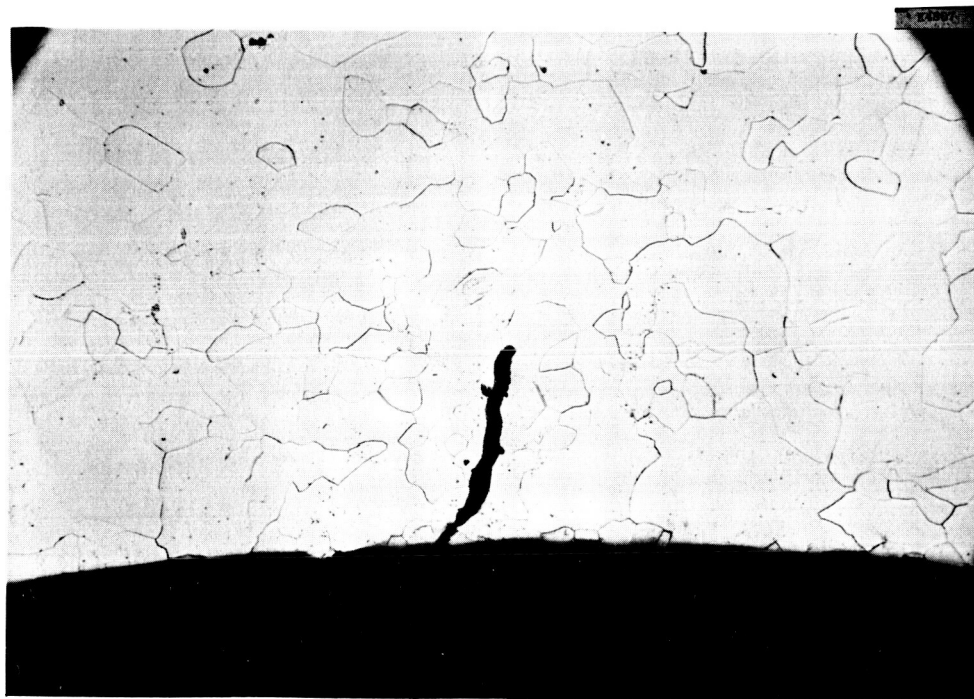
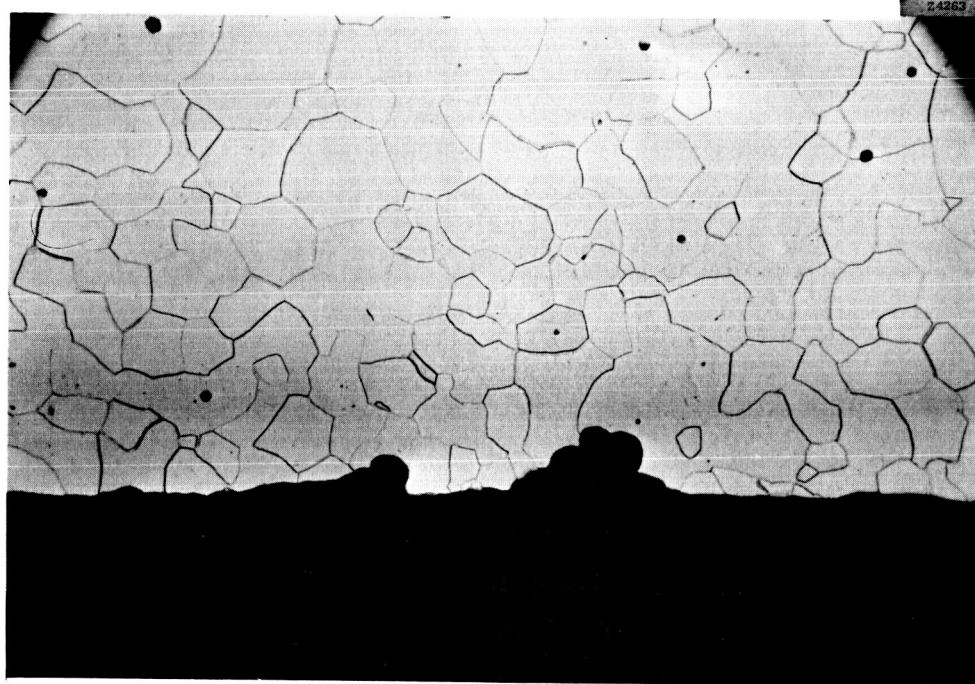


Figure 3. Typical ID Defects Observed in Cb-1Zr Alloy 0.375-Inch OD x 0.065-Inch Thick Wall Tubes. Top: Outer Surface of MCN 404-4 (Z4263); Bottom: Inner Surface of MCN 447 (K4807).

Etchant: 20%HF-20%HNO₃-60%Water

Mag: 100X

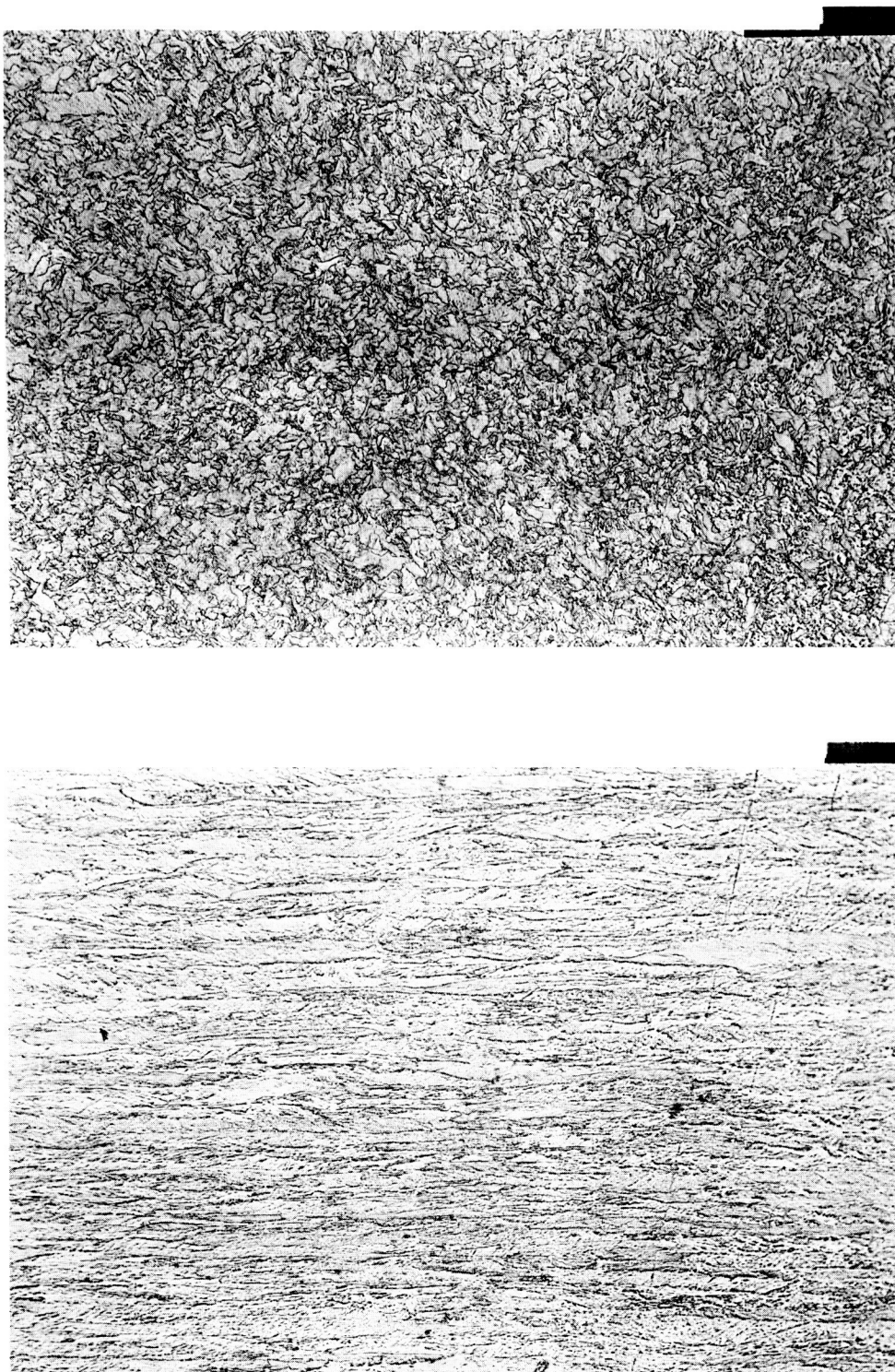


Figure 4. Microstructure of Mo-TZM Alloy One-Inch Diameter Bar (MCN 435) Used in the Fabrication of the Turbine Simulator for the Prototype Loop. Final Heat Treatment was 3/4 Hour at 2300°F. Top: Transverse Section (K1003); Bottom: Longitudinal Section (K1004).

Etchant: Murakami's

Mag: 100X

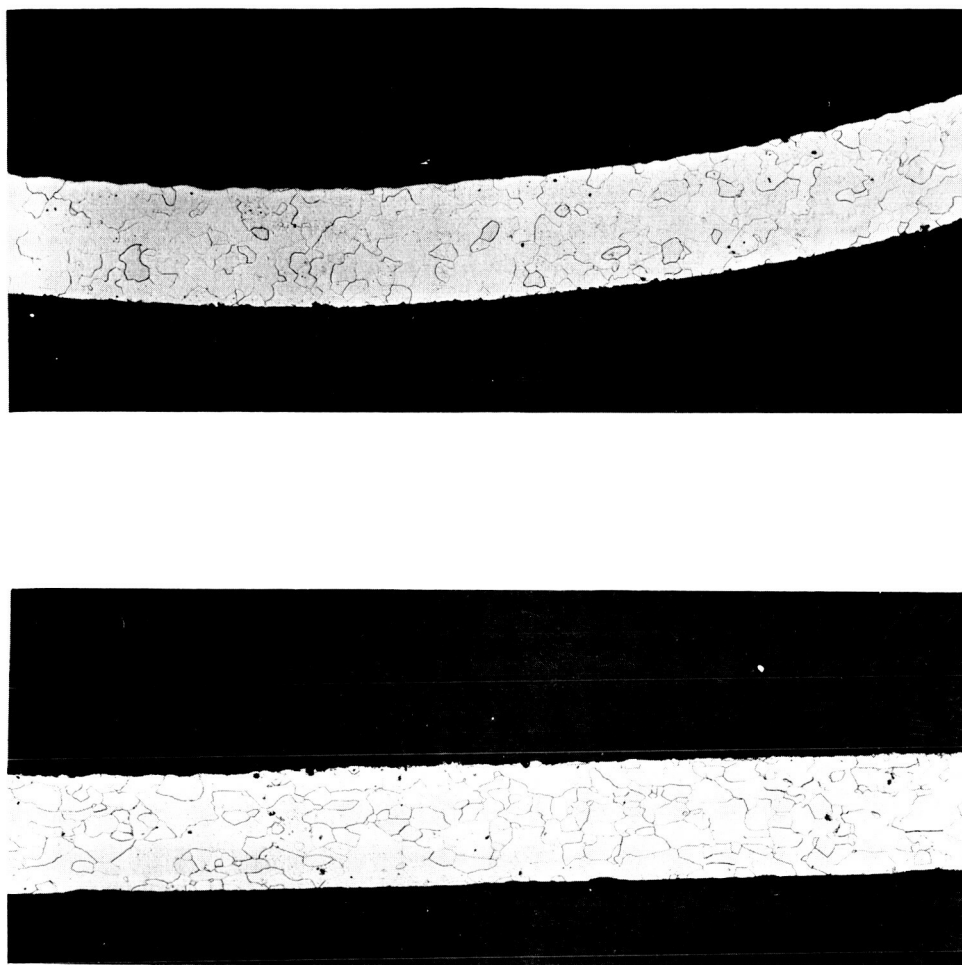


Figure 5. Microstructure of Cb-1Zr Alloy 0.375-Inch OD x 0.008-Inch Thick Wall Tube (MCN 466) Produced by Wolverine Tube Division. Final Heat Treatment was One Hour at 2200°F. Top: Transverse Section (K5214); Bottom: Longitudinal Section (K5215).

Etchant: 20%HF-20%HNO₃-60%Water

Mag: 100X

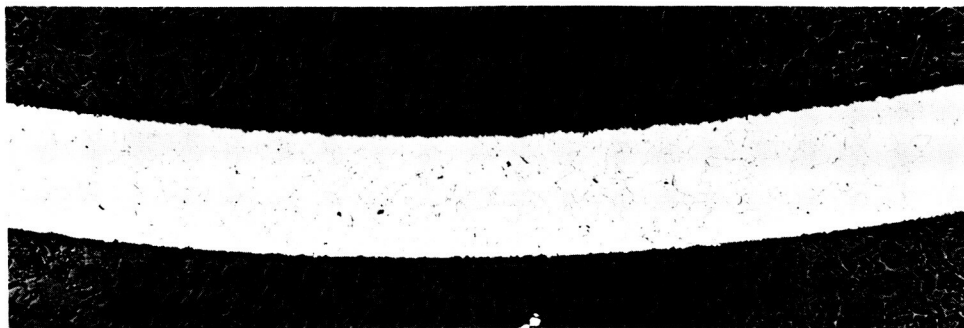


Figure 6. Microstructure of T-111 Alloy 0.375-Inch OD x 0.008-Inch Thick Wall Tube (MCN 509) Produced by National Research Corporation, Metals Division. Final Heat Treatment was 1.5 Hours at 2700°F. Top: Transverse Section (L1287); Bottom: Longitudinal Section (L1288).

Etchant: 20%HF-20%HNO₃-60%Water

Mag: 100X



Figure 7. Microstructure of D-43 Alloy 0.375-Inch OD x 0.008-Inch Thick Wall Tube Produced by Wolverine Tube Division. Final Heat Treatment was One Hour at 2850°F. Top: Transverse Section Lot A (MCN 491--Best Quality); Bottom: Transverse Section Lot B (MCN 492--Poorest Quality).

Etchant: 20%HF-20%HNO₃-60%Water

Mag: 100X

APPENDIX A
REFRACTORY ALLOY MATERIAL SPECIFICATIONS

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01-0003-02-B
SPPS-1B
30 August 1963
Page 1 of 16

SPECIFICATION

BAR, ROD, SHEET, PLATE AND STRIP:

COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

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GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

BAR, ROD, SHEET, PLATE, AND STRIP: COLUMBIUM - 1% ZIRCONIUM ALLOY

1. SCOPE.

1.1. Scope. This specification covers a wrought reactor grade columbium - 1% zirconium alloy in bar, rod, sheet, plate, and strip form.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents

ASTM Designation B
(Tentative)
(23 August 1961)

Columbium Alloy Ingots

ASTM Designation E8-57T
(26 December 1957)

Methods of Tension Testing of
Metallic Materials

ASTM Designation
(Pending)

Methods for Chemical Analysis of
Reactor and Commercial Columbium

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspection

AMS 2646
(1 March 1955)

Contrast Dye Penetrant
Inspection

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and Heat
Resistant Sheet, Strip, and Plate

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots conforming to ASTM Designation B, tentative specification for columbium alloy ingots. These ingots may be produced by fusion, powder metallurgy methods, or other suitable means which will allow processing the consolidated metal into various basic shapes.

3.3. Chemical Composition

3.3.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I. (Refer to page 4.)

3.3.2. Final Product Composition. The manufacturer's ingot analysis shall be considered the chemical analysis for products supplied under this specification except carbon, oxygen, nitrogen, and hydrogen content, to conform to the requirements in Table I, shall be determined in the finished product. (Refer to Table I, page 4.)

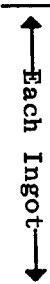
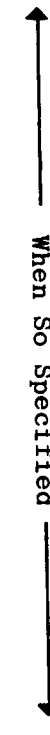
3.3.3. Check Analysis. Upon check analysis final product compositions shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max. (ppm)</u>	<u>Permissible Variations in Check Analysis (ppm)</u>
Carbon	100	+ 10
Oxygen	300	+ 50
Nitrogen	300	+ 50
Hydrogen	15	+ 2

3.4. Condition

3.4.1. Processing. Unless otherwise stated by the purchaser, after a final cold reduction of 20-40% the finished product shall be annealed in a vacuum for one hour at 2200°F. Before this final 20-40% reduction, regardless of the previous inprocess anneals, the semifinished product shall be vacuum annealed for one hour at 2200°F. As an alternate, vacuum annealing at 1800°F for one hour before the final 20-40% cold reduction is permissible, provided the material has been cold-reduced at least 75% after the previous inprocess anneal.

TABLE I
CHEMICAL COMPOSITION
REACTOR GRADE Cb-1Zr

<u>Element</u>	<u>Maximum Content</u> <u>(ppm)</u>	<u>Analysis</u> <u>Requirements</u>
Carbon	100	
Nitrogen	300	
Oxygen	300	
Hydrogen	15	
Zirconium	0.8 - 1.2% (range)	
Iron	500	
Tantalum	1000	
Titanium	500	
Silicon	300	
Boron	2	
Tungsten	500	
Molybdenum	1000	
Aluminum	---	
Beryllium	---	
Cadmium	5	
Chromium	---	
Cobalt	30	
Copper	---	
Lead	50	
Lithium	---	
Magnesium	---	
Manganese	100	
Nickel	200	
Tin	---	
Vanadium	200	
Ytterbium	---	
Zinc	---	
Uranium	---	
Hafnium	100	
Calcium	---	
Sodium	---	
Chlorine	---	
Columbium, by difference	98.5% min	

3.4.2. Microhardness. After heat treatment the material shall be sectioned and a representative sample shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat-treating atmosphere. A microhardness traverse shall not show a hardness gradient over 50 VHN points from the center to the surface of a cross-sectional sample of the material.

3.4.3. Grain Size. Unless otherwise agreed to by the purchaser and the vendor, the grain size of the finished product shall be such that the length of the grains parallel to the working direction shall not exceed 0.05 inch or the smallest dimension of the product, whichever is less; the grain size perpendicular to the working direction shall be smaller than that indicated by ASTM standard grain size No. 3.

3.5. Mechanical Properties

3.5.1. Room Temperature Tensile Strength. Longitudinal specimens (or transverse where practical) shall not exceed the following property limits at test temperatures of 65° - 85°F:

<u>Tensile Strength</u> <u>(psi)</u>	<u>Yield Strength</u> <u>(0.2%, psi)</u>	<u>Elongation</u> <u>(2-inch gauge, %)</u>
75,000 max	60,000 max	10 min

3.5.2. Stress-to-Rupture Test. The material in final form shall be capable of achieving the following stress-rupture life under suitable environmental conditions:

<u>Test Temp</u> <u>(°F)</u>	<u>Stress</u> <u>(psi)</u>	<u>Minimum Life</u> <u>(hours)</u>
2000	10,000	25

After the stress-rupture test, check analysis of the specimen shall not exceed the following limits:

<u>Element</u>	<u>Check Analysis Limits</u> <u>(ppm)</u>
Carbon	150
Oxygen	400
Nitrogen	400
Hydrogen	15

3.5.3. Hardness. The product shall have a Rockwell hardness number not exceeding B-90 in the heat-treated condition.

3.6. Tolerances

3.6.1. Rolled, Swaged, or Drawn Rounds

3.6.1.1. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter (inches)</u>	<u>Diameter Variation (inch)</u>	<u>Out-of-Roundness (inch)</u>
.020 to .030	+ .0005 - .0005	
.030 to .062	+ .001 - .001	
1/16 to 9/32	+ .002 - .002	.004
Over 9/32 to 13/32	+ .010 - .005	.008
Over 13/32 to 5/8	+ .010 - .055	.012
Over 5/8 to 7/8	+ .015 - .005	.015
Over 7/8 to 1	+ .020 - .005	.015
Over 1 to 1-3/8	+ .020 - .010	.018
Over 1-3/8 to 1-1/2	+ .020 - .015	.020
Over 1-1/2 to 1-5/8	+ .025 - .015	.020
Over 1-5/8 to 2	+ .030 - .020	.025
Over 2 to 2-1/2	+ .032 - .032	.025
Over 2-1/2 to 3-1/4	+ .032 - .032	.027
Over 3-1/4 to 3-1/2	+ .045 - .045	.040
Centerless ground rounds		
1/16 to 2	+ .002 - .002	
Over 2	+ .003 - .002	

3.6.1.2. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.6.1.3. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.6.2. Square or Rectangular Bar or Plate

3.6.2.1. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 10%	± 0.125 inch	± 0.125 inch

3.6.2.2. Flatness of Plate. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip, and Plate."

3.6.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.6.3. Sheet or Strip

3.6.3.1. Thickness. The permissible variations in thickness shall not exceed those presented in Table III (1/2 the tolerance values in AMS2242A). Measurements shall be taken 1 inch in from the edges on all sheets at the corners and mid-sides to the nearest 0.0001 inch.

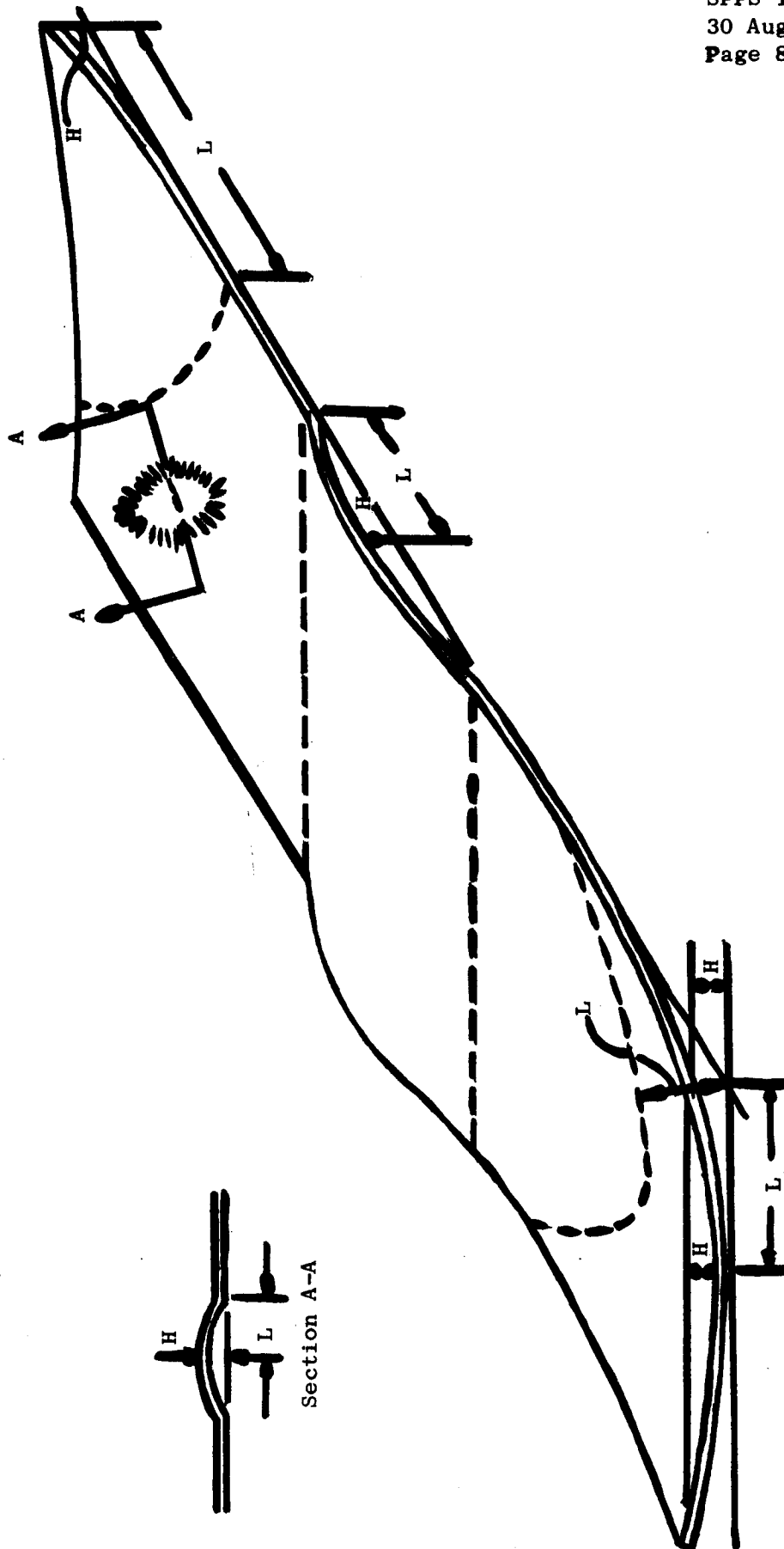
TABLE III
THICKNESS TOLERANCES

<u>Nominal Gauge</u> <u>(inch)</u>	<u>Tolerance</u> <u>(inch, plus or minus)</u>
.005	.0005
Over .005 to .007 inclusive	.00075
Over .007 to .016 inclusive	.001
Over .016 to .026 inclusive	.0015
Over .026 to .040 inclusive	.002
Over .040 to .058 inclusive	.0025
Over .058 to .072 inclusive	.003
Over .072 to .083 inclusive	.0035
Over .083 to .098 inclusive	.004
Over .098 to .114 inclusive	.0045
Over .114 to .130 inclusive	.005
Over .130 to .145 inclusive	.006
Over .145 to .187 inclusive	.007

3.6.3.2. Length. Maximum deviation shall be 0.125 inch.

3.6.3.3. Width. Maximum deviation shall be 0.064 inch.

3.6.3.4. Flatness. Flatness tolerances shall be 4%, determined by the method shown in Figure 1. (Refer to Page 8). The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.) of these defects shall be made.



H = Maximum distance between flat surface and lower surface of sheet.

L = Minimum distance between highest point on sheet and point of contact with flat surface.

—line of tangency between sheet and flat surface.

$H/L \times 100 = \% \text{ flatness}$

Figure 1. Method for Determining Flatness in Sheet and Strip.

3.7. Maximum Allowable Discontinuities

3.7.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

3.7.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of thickness, whichever is smaller, shall be a minimum of 0.050 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

3.7.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections shall be faired smooth to remove any notch effect.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certification. The analysis made by the material manufacturer to determine the percentage of elements stipulated in this specification shall be reported to the purchaser in a specified certificate of test.

4.1.2. Methods of Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium".

4.2. Tensile Test

4.2.1. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.2.2. Test Methods. The tension test shall be conducted in accordance with "Methods of Tension Testing of Metallic Materials," ASTM Designation E8-57T. Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a free crosshead speed of 0.05 plus 0.02 inch per inch of gauge length per minute throughout the test.

4.3. Stress-Rupture Test

4.3.1. Necessity of Test. When agreed between the purchaser and the vendor, stress-rupture properties of specimens from each heat of material shall be determined by mutually acceptable testing techniques.

4.3.2. Test Techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 10^{-6} torr or better.

Specimens shall be wrapped in tantalum or zirconium foil to minimize contamination.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

4.4. Number of Tests Required. Representative test specimens from each lot or ingot of material, whichever is larger, shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finish product chemistry - one per lot or ingot
Tensile test - two per lot or ingot
Stress-rupture test - two per lot or ingot.

4.5. Retest and Rework

4.5.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

4.5.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

4.6. Inspection

4.6.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.6.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

4.6.3. Methods of Inspection

4.6.3.1. Radiographic. Whenever specified, the product shall be radiographed and determined sound as described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

4.6.3.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

4.6.3.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse. For round bar and rod, inspection shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). For bar and rod, automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified. A reflector may be used for thin sheet, if desired, but is not required.

4.6.3.2.2. Calibration of Bar, Rod, and Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the plate thickness, 1.5% of the bar or rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface at a distance from the

surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$, and $3/4$ the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on plate with shear wave the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

4.6.3.2.3. Calibration of Sheet and Strip. Calibration for longitudinal beam shall be accomplished by setting some convenient multiple or reflected signal at 100% after having established that the beam is perpendicular to the surface. Sensitivity shall be assured by detecting an 0.086-inch diameter flatbottom hole drilled approximately half way through the thickness of the sheet.

The sheet shall also be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scraped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125 inch wide in its smaller dimension (where it enters the sheet being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration sheet; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). The calibration notch shall be detected when it is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which

produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all sheet material inspected since then shall be reinspected.

4.6.3.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on plate with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

4.6.3.2.5. Reporting. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

4.6.3.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor. Inspection results shall be reported to the purchaser.

4.6.3.3. Penetrant Inspection. Whenever specified, the exterior surface of the product shall be penetrant inspected and found free of flaws using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

4.7. Reports. The manufacturer shall supply at least three copies of a report showing test results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.8. Rejection. Material not conforming to this specification or to authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. All material shall be packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS.

6.1. Rod. Any straight product with a round, hexagonal, or octagonal solid cross section, 3 1/2 inches in diameter or less, or equivalent cross section.

6.2. Bar. Any straight product with a rectangular cross section 0.187 inch or more thick and less than 5 inches wide, or with a square cross section not conforming to the definition of wire.

6.3. Plate. Any product over 0.187 inch thick and 5 or more inches wide.

6.4. Sheet. Any product under 0.187 inch thick and 5 or more inches wide.

6.5. Strip. Any product less than 0.187 inch thick and less than 5 inches wide.

6.6. Lot. All material of the same size, shape, condition, and finish on an order produced from one or more ingots and annealed in the same vacuum annealing charge.

6.7. Check Analysis. An analysis made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

6.8. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 1/2 hour

NEW SPEC. NO. ASSIGNED

01-0003-02-B

SPEC. NOT REVISED

4-1-65

SPPS-1B

Amendments

30 August 1963

Page 16 of 16

Amendment No. 1. Change paragraphs 3.3.1. through 3.3.3. and Table I of SPPS-1B to limit the hydrogen content to 10 ppm, maximum.

Amendment No. 2. Change paragraph 3.5.2. of SPPS-1B to require that the chemical analyses of the stress-rupture specimen after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

Amendment No. 3. Add to paragraph 3.4.1. of SPPS-1B the requirement that all inprocess annealing and final annealing shall be conducted in a vacuum with the pressure less than 1.0×10^{-4} torr.

Amendment No. 4. Add to paragraph 3.4.1. of SPPS-1B the requirement that the semifinished and finished Cb-1Zr products shall be wrapped with two layers of 0.002 to 0.005-inch thick, tantalum, columbium, or Cb-1Zr foil for the purpose of providing additional protection from contamination during annealing in a vacuum (pressure less than 1.0×10^{-4} torr).

SPECIFICATION

BAR, ROD, SHEET, PLATE AND STRIP: COLUMBIUM - 1% ZIRCONIUM

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

BAR, ROD, SHEET, PLATE AND STRIP:
COLUMBIUM - 1% ZIRCONIUM

- CONTINUED

DATE

4-19-65

NO.

01-0003-03-B

1. Only the following paragraphs of specification no. 01-0003-02-B are applicable:

3.1.
3.2.
3.3.
3.3.1.
3.3.2.
3.4.1. (Final Anneal Only)
3.7.1.
3.7.3.

2. Only amendments 1, 3 and 4 are applicable.

All items shall be clearly identified with the revised specification number.

01-0003-04-B
19 April 1965
Page 1 of 2

SPECIFICATION

BAR, ROD, SHEET, PLATE AND STRIP: COLUMBIUM-1% ZIRCONIUM

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

BAR, ROD, SHEET, PLATE AND STRIP:
COLUMBIUM-1% ZIRCONIUM

- CONTINUED

DATE

4-19-65

NO.

01-0003-04-B

The following exceptions to specification no. 01-003-02-B are applicable.

Paragraph 3.3.1. Ingot/Billet Composition is changed to read: The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page four). A minimum of four analyses shall be obtained as follows: ingot/billet top-center, mid-radius and edge; ingot/billet bottom-center. All analyses must conform to ranges stated in Table I.

Paragraph 3.5.2. Stress-Rupture requirement is deleted.

Paragraph 4.3. Stress-Rupture requirement is deleted.

Paragraph 4.4. Number of Tests required is changed to read:

Finished Product Chemistry - one per lot per ingot
Tensile Test - two per lot per ingot
Grain Size - two per lot per ingot
Microhardness Traverse - one per lot per ingot

Paragraph 4.6.3.1. Radiographic requirement is deleted.

Paragraph 4.6.3.3. Penetrant Inspection is specified.

Paragraph 6.6. Lot is changed to read: All material of the same size, shape, condition and finish produced from one ingot and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which the temperature may reach 500°F or above. When processing temperatures are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical and tensile tests only, provided prior written approval has been obtained from the purchaser.

Only amendments 1, 3 and 4 are applicable.

All items shall be clearly identified with the revised specification number.

SPECIFICATION

SEAMLESS TUBING: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

SEAMLESS TUBING: COLUMBIUM - 1% ZIRCONIUM ALLOY

1. SCOPE

1.1. Scope. This specification covers seamless tubing of reactor grade columbium - 1% zirconium alloy.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents

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(23 August 1961)

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3.2. Manufacture. Material covered by this specification shall be made from ingots conforming to ASTM Designation B, tentative specification for columbium alloy ingots. These ingots may be produced by fusion, powder metallurgy methods, or other suitable means which will allow processing the consolidated metal into various basic shapes.

3.3. Chemical Composition

3.3.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I. (Refer to page 4.)

3.3.2. Final Product Composition. The manufacturer's ingot analysis shall be considered the chemical analysis for products supplied under this specification; except carbon, oxygen, nitrogen, and hydrogen content shall be determined in the finished products and shall conform to the requirements in Table I. (Refer to page 4.)


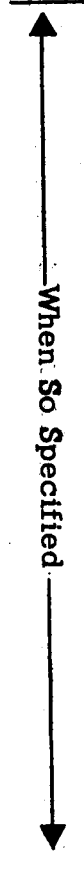
3.3.3. Check Analysis. Upon check analysis final product compositions shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max, (ppm)</u>	<u>Permissible Variations in Check Analysis (ppm)</u>
Carbon	100	+ 10
Oxygen	300	+ 50
Nitrogen	300	+ 50
Hydrogen	15	+ 2

3.4. Condition

3.4.1. Processing. Unless otherwise stated by the purchaser, after a final reduction of 20-40% the finished product shall be annealed for one hour at 2200°F in a vacuum. Before the final 20-40% reduction the semifinished product shall be vacuum annealed for one hour at 2200°F regardless of the previous inprocess anneals. As an alternate, vacuum annealing at 1800°F for

TABLE I
CHEMICAL COMPOSITION
REACTOR GRADE Cb-1Zr

<u>Element</u>	<u>Maximum Content (ppm)</u>	<u>Analysis Requirements</u>
Carbon	100	
Nitrogen	300	
Oxygen	300	
Hydrogen	15	
Zirconium	0.8 - 1.2% (range)	
Iron	500	
Tantalum	1000	
Titanium	500	
Silicon	300	
Boron	2	
Tungsten	500	
Molybdenum	1000	
Aluminum	--	
Beryllium	--	
Cadmium	5	
Chromium	--	
Cobalt	30	
Copper	--	
Lead	50	
Lithium	--	
Magnesium	--	
Manganese	100	
Nickel	200	
Tin	--	
Vanadium	200	
Ytterbium	--	
Zinc	--	
Uranium	--	
Hafnium	100	
Calcium	--	
Sodium	--	
Chlorine	--	
Columbium, by difference	98.5% min	

on hour before the final 20-40% cold reduction is permissible provided the material has been cold-reduced at least 75% after the previous inprocess anneal.

3.4.2. Microhardness. After heat treatment the material shall be sectioned and a representative sample shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat-treating atmosphere. A microhardness traverse shall not show a hardness gradient over 50 VHN points from the center to the surface of a cross-sectional sample of the material.

3.4.3. Grain Size. Unless otherwise agreed to by the purchaser and the vendor, the grain size of the finished product shall be such that at least thirty grains are intersected by a traverse through the wall thickness of the tube, and the length of grains, in the direction of the tube length, shall not exceed 0.05 inch or the wall thickness of the tube, whichever is smaller.

3.5. Mechanical Properties

3.5.1. Room Temperature Tensile Strength. The following property limits shall not be exceeded on longitudinal specimens (or transverse where practical) at test temperatures of 65°F - 85°F.

<u>Tensile Strength</u> <u>(psi)</u>	<u>Yield Strength</u> <u>(0.2%, psi)</u>	<u>Elongation</u> <u>(2-inch gauge, %)</u>
75,000 max	60,000 max	10 min

3.5.2. Stress-to-Rupture Test. The material in final form shall be capable of achieving the following stress-rupture life under suitable environmental conditions:

<u>Test Temp</u> <u>(°F)</u>	<u>Stress</u> <u>(psi)</u>	<u>Minimum Life</u> <u>(hours)</u>
2000	10,000	25

After the stress-rupture test check analyses of the specimen shall not exceed

the following limits:

<u>Element</u>	<u>Check Analysis Limits</u> <u>(ppm)</u>
Carbon	150
Oxygen	400
Nitrogen	400
Hydrogen	15

3.5.3. Hydrostatic Test. When agreed to by the purchaser and the vendor, each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

- P = hydrostatic test pressure in pounds per square inch;
- S = 12,000 psi;
- t = average wall thickness of the tube in inches;
- D = outside diameter of the tube in inches.

3.5.4. Flare Test. A section of the heat-treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.5.5. Hardness Tests. Rockwell hardness tests shall be made on the inside of a specimen cut from the tubes having a wall thickness greater than 0.015 inch. The tube shall have a Rockwell hardness number not exceeding B-90 in the heat-treated condition.

3.6. Tolerances

3.6.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II. (Refer to page 7 .)

3.6.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet a variation of plus 1/4 inch will be permitted, unless otherwise specified.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD ⁽¹⁾ (inches)	OD ⁽²⁾ (inch)	ID ⁽³⁾ (inch)	Wall Thickness ⁽⁴⁾ (%)
0.187 to but not incl 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl 4.000	± 0.0125	± 0.0125	± 10

- (1) Tolerances are applicable to only two dimensions, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010 inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

3.6.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.7. Maximum Allowable Discontinuities.

3.7.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

3.7.2. Porosity and Inclusions. Indications with diameters greater than 0.020 inch or 3% of the wall thickness, whichever is the smaller, shall be unacceptable. Indications with diameters in the range of 0.010 inch to 0.020 inch or 1% to 3% of wall thickness, whichever is smaller, must be a minimum of 0.50 inch apart; those indications with dimensions less than 0.010 inch must be a minimum of 0.12 inch apart.

3.7.3. Surface Rework. Defects less than 3% of the nominal wall thickness may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II. (Refer to page 7.)

4. QUALITY ASSURANCE PROVISIONS.

4.1. Chemical Analysis.

4.1.1. Certification. The analysis made by the material manufacturer to determine the percentage of elements stipulated in this specification shall be reported to the purchaser in a specified certificate of test.

4.1.2. Methods of Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium".

4.2. Tensile Test.

4.2.1. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.2.2. Test Methods. The tension test shall be performed in accordance with "Methods of Tension Testing of Metallic Materials," ASTM Designation E8-57T. Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a free crosshead speed of 0.05 plus 0.02 inch per inch of gauge length per minute throughout the test. The specimen, whenever possible, shall be a full cross section.

4.3. Stress-Rupture Test

4.3.1. Necessity of Test. When agreed between the purchaser and the vendor, stress-rupture properties of specimens from each heat of material shall be determined by testing according to mutually agreeable techniques.

4.3.2. Test Techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 10^{-6} torr or better.

Specimens shall be wrapped in tantalum or zirconium foil to minimize contamination.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

4.4. Number of Tests Required. Representative test specimens from each lot or ingot of material, whichever is larger shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

- : Finish product chemistry - one each per lot or ingot
- : Tensile test - two per lot or ingot
- : Stress-rupture test - two per lot or ingot
- : Flare test - two per lot or ingot
- : Hydrostatic proof test - 100%.

4.5. Retest and Rework

4.5.1. Surface Contamination. Any sample or specimen exhibiting obvious

surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

4.5.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

4.6. Inspection

4.6.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.6.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

4.6.3. Methods of Inspection

4.6.3.1. Radiographic. When specified, the product shall be radiographed and determined sound as described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on particular product.

4.6.3.2. Penetrant Inspection. When specified, the exterior surface of the product shall be penetrant inspected and found free of flaws using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

4.6.3.3. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

4.6.3.3.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by longitudinal wave and by both circumferential and axial shear techniques, unless otherwise specified. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

4.6.3.3.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 10%), all inspections since the previous calibration shall be repeated.

4.6.3.3.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter calibration notch. Defects whose amplitude is 60% or greater shall be reported giving axial and circumferential location and distance from the surface.

4.7. Reports. If requested, the vendor shall supply at least three copies of a report which shows the results of tests performed on each lot of material in the shipment and includes the number of the specification and the purchase order or contract number.

4.8. Rejection. Material not conforming to this specification or to authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. All material shall be packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. All material of the same size, shape, condition, and finish on an order produced from one or more ingots and annealed in the same vacuum annealing charge.

6.2. Check Analysis. An analysis either made or requested by the purchaser of the metal after it has been processed into finished mill forms for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

6.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed

value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 1/2 hour

NEW SPEC. NO. ASSIGNED

01-0004 - 02-B
SPEC. NOT REVISED

4-1-65

SPPS-2B

Amendments

30 August 1963

Page 14 of 14

Amendment No. 1. Change paragraphs 3.3.1 through 3.3.3 and Table I of SPPS-2B to limit the hydrogen content to 10 ppm, maximum.

Amendment No. 2. Change paragraph 3.5.2 of SPPS-2B to require that the chemical analyses of the stress-rupture specimen after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

Amendment No. 3. Add to paragraph 3.4.1 of SPPS-2B the requirement that all inprocess annealing and final annealing shall be conducted in a vacuum with the pressure less than 1.0×10^{-4} torr.

Amendment No. 4. Add to paragraph 3.4.1 of SPPS-2B the requirement that the semifinished and finished Cb-1Zr products shall be wrapped with two layers of 0.002 to 0.005-inch thick, tantalum, columbium, or Cb-1Zr foil for the purpose of providing additional protection from contamination during annealing in a vacuum (pressure less than 1.0×10^{-4} torr).

01-0004-03-B
19 April 1965
Page 1 of 2

SPECIFICATION

SEAMLESS TUBING: COLUMBIUM - 1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SEAMLESS TUBING: COLUMBIUM - 1% ZIRCONIUM ALLOY	DATE 4-19-65	NO. 01-0004-03-B
CONTINUED		

The following exceptions to specification no. 01-0004-02-B are applicable:

Paragraph 3.3.1. Ingot/Billet Composition is changed to read: The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page four). A minimum of four analyses shall be obtained as follows: ingot/billet top-center, mid-radius and edge; ingot/billet bottom-center. All analyses must conform to ranges stated in Table I.

Paragraph 3.5.2. Stress-Rupture requirement is deleted.

Paragraph 4.3. Stress-Rupture requirement is deleted.

Paragraph 4.4. Number of Tests Required is changed to read:

Finished Product Chemistry - one per lot per ingot
Tensile Test - two per lot per ingot
Flare Test - two per lot per ingot
Grain Size - two per lot per ingot
Microhardness Traverse - one per lot per ingot
Hydrostatic Proof Test - 100%

Paragraph 4.6.3.1. Radiographic requirement is deleted.

Paragraph 4.5.3.3. Penetrant Inspection is specified.

Paragraph 6.1. Lot is changed to read: All material of the same size, shape, condition and finish produced from one ingot and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which the temperature may reach 500°F or above. When processing temperatures are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical and tensile tests only, provided prior written approval has been obtained from the purchaser.

Only amendments 1, 3 and 4 are applicable.

All items shall be clearly identified with the revised specification number.

01-0009+00-B
SPPS-15-R1
23 December 1964
Page 1 of 14

SPECIFICATION

BAR AND ROD: Mo-TZM (Mo-0.5Ti-0.08Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: Mo-TZM (Mo-0.5Ti-0.08Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers Mo-TZM (Mo-0.5Ti-0.08Zr) alloy in bar and rod form intended for high temperature structural applications in alkali metal systems.

1.2. <u>Classes</u> .	SPPS-15A-R1	Recrystallized
	SPPS-15B-R1	Stress-Relieved

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Methods of Tension Testing of Metallic Materials
ASTM Designation E29-58T	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspection
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction and in-process and final annealing, aging or stress-relieving temperatures and times shall be selected by the vendor to achieve the structure or grain size range specified in paragraph 3.6. and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final products of Classes A and B shall exceed 75%; the amount of final reduction imparted to mill products of Class B since the last recrystallization anneal and prior to the final stress-relief heat treatment shall not exceed 80%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. Class A. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Class B. The finished product shall be supplied in the stress-relieved condition throughout the cross-sectional area as specified in paragraph 3.6.

3.4.3. Heat Treating. All heating shall be carried out in a vacuum less than 1×10^{-4} torr or in a hydrogen atmosphere with a dew point of less than -100°F . All mill products to be heat treated shall be thoroughly degreased, chemically cleaned and protected from the furnace parts by an appropriate refractory metal layer. The conditions of final heat treating shall be reported in the certificate of compliance.

3.4.4. Surface Contamination. All items are to be free of contamination or internal oxidation determined by the following technique: representative samples of each bar diameter shall be subjected to thermal treatment sufficient to induce approximately 80% recrystallization throughout the core of the cross-sectional area. Indications of inhibited recrystallization at or near the surface by metallographic examination of transverse sections of heat treated samples shall be

construed as contamination and be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimension and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100 min; 400 max	± 50
Oxygen	25	+ 5
Nitrogen	15	+ 2
Hydrogen	5	+ 2

3.6. Structure

3.6.1. Class A. The grain size of the final products shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	100
Over 0.250 to 0.500	4	2	100
Over 0.500 to 1.0	4	2	100
Over 1.0 to 2.0	4	2	95
Greater Than 2.0	3	3	90

TABLE I
CHEMICAL COMPOSITION
Mo-TZM (Mo-0.5Ti-0.08Zr) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>w/o</u>	<u>Maximum Content</u> <u>w/o</u>	<u>Analysis</u> <u>Required</u>
Molybdenum	99.25	---	↑ Each Ingot
Titanium	0.40	0.55	
Zirconium	0.08	0.12	
Carbon	0.01	0.04	
		<u>ppm</u>	
Oxygen	---	20	↑ When so Specified ↓
Nitrogen	---	10	
Hydrogen	---	5	
Tungsten	---	120	
Silicon	---	80	
Iron	---	80	
Chromium	---	25	
Tin	---	40	
Nickel	---	20	
Copper	---	20	
Aluminum	---	20	
Calcium	---	20	
Manganese	---	20	
Magnesium	---	20	
Cobalt	---	20	
Lead	---	10	
Tantalum	---	100	
Columbium	---	100	
Vanadium	---	100	

3.6.2. Class B. The grain size of the final product shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable Equivalent ASTM Grain Size No. Perpendicular to Direction of Work</u>	<u>% R_x Maximum</u>
0.125 to 0.250	4	5
Over 0.250 to 0.500	4	5
Over 0.500 to 1.0	4	5
Over 1.0 to 2.0	4	5
Greater Than 2.0	3	5

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Class</u>	<u>Diameter or Maximum Distance Between Parallel Sides, Inches</u>	<u>Minimum Ultimate Tensile Strength, ksi</u>	<u>Minimum 0.2% Yield Strength, ksi</u>	<u>Minimum Elong., % in 4D</u>
A	Less Than 2.0	80	55	20
A	Over 2.0 to 4.5	75	45	10
B	0.125 to 0.875	115	100	18
B	Over 0.875 to 1.125	110	95	15
B	Over 1.125 to 1.875	100	85	10
B	Over 1.875 to 2.875	90	80	10
B	Over 2.875 to 3.500	85	75	5
B	Over 3.500 to 4.500	80	70	5

3.7.2. Room Temperature Hardness. Rod and bar supplied under this specification shall conform to the following hardness requirements when tested transverse to the final rolling direction.

<u>Diameter or Maximum Distance Between Parallel Sides, Inches</u>	<u>Hardness Range (DPH - 10 Kg. Load)</u>
0.125 to 0.875	260-320
Over 0.875 to 1.125	250-310
Over 1.125 to 1.875	245-300
Over 1.875 to 2.875	240-290
Over 2.875 to 3.500	235-285
Over 3.500 to 4.500	230-280

3.7.3. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Class</u>	<u>Test Temp. °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
A	2200	25	20
B	2200	40	20

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter</u> <u>Inches</u>	<u>Diameter</u> <u>Variation</u> <u>Inch</u>	<u>Out-of-Roundness</u> <u>Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.020	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040

Centerless Ground Rounds

0.0625 to 2.00	+ 0.002, -0.002
Over 2.00	+ 0.003, -0.002

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$, whichever is less	± 0.125 Inch	± 0.125 Inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2 Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. The inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum of material, i.e., specimens from material of Class A may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes may be settled by accepted referee methods.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined by using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-5} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in

paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005-inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of 1/2 the thickness or diameter or, if the thickness exceeds 0.750 inch, 1/4, 1/2 and 3/4 of thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at 1/4 and 1/2 the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and locations of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification.

purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box, or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

01-0009-01-B
20 April 1965
Page 1 of 2

SPECIFICATION

BAR AND ROD: Mo-TZM (Mo-0.5Ti-0.08Zr) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SP 1073 A

BAR AND ROD: Mo-TZM (Mo-0.5Ti-0.08Zr) ALLOY - CONTINUED	DATE 4-20-65	NO. 01-0009-01-B
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The following exception of Specification No. 01-0009-00-B is:

Table I - Chemical Composition: The minimum zirconium content is lowered to 0.06 weight per cent.

SPECIFICATION

BAR AND ROD: T-111 (Ta-8W-2Hf) ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

BAR AND ROD: T-111(Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Des- ignating Significant Places in Specified Limiting Values
ASTM Designation E112-61 (1961)	Estimating the Average Grain Size of Metals
ASM 2635 (15 August 1958)	Radiographic Inspection
ASM 2645 (1 March 1955)	Fluorescent Penetrant Inspec- tion
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspec- tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 4D</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3.).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: Total increase in oxygen plus nitrogen content -- less than 50 ppm; increase in hydrogen content -- less than 5 ppm; increase in carbon content -- less than 10 ppm. The following limits shall apply to the analytical results:

Carbon \pm	10 ppm
Oxygen \pm	25 ppm
Nitrogen \pm	25 ppm
Hydrogen \pm	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25 inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$, whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

TABLE II
PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter Inches</u>	<u>Diameter Variation Inch</u>	<u>Out-of-Roundness Inch</u>
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	+ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.020	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040

Centerless Ground Rounds

0.0625 to 2.0	+ 0.002, -0.002
Over 2.0	+ 0.003, -0.002

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from bar of sufficient width or from rod greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset, and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater

than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ of thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750 inch, shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitude and gain settings, and the amplitude and locations of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in a heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

01-0035-00-B
SPPS-54-R1
23 December 1964
Page 1 of 14

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in tube and pipe form intended for high temperature structural application and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspection

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment, and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	75	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.0125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %⁽¹⁾</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analyses of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	\pm	10 ppm
Oxygen	\pm	25 ppm
Nitrogen	\pm	25 ppm
Hydrogen	\pm	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600; unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the ranges of 1% to 3% of wall thickness must be a minimum of 0.50-inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all test and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement of purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operation.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraph 5.4.2. and 5.4.3 shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing technique for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared

to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacture shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within in a heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right- hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SHEET, PLATE, AND STRIP: T-111 (Ta-8W-2Hf) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SHEET, PLATE, AND STRIP: T-111 (Ta-8W-2Hf) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-111 (Ta-8W-2Hf) alloy in sheet, plate and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating the Average Grain
Size of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and
Heat Resistant Sheet, Strip
and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for
Refractory Metal Sheet Mate-
rials

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing

conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined in the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	50	+ 10
Oxygen	150	+ 20
Nitrogen	75	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE I
CHEMICAL COMPOSITION
T-111 (Ta-8W-2Hf) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	-	50
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., % in 2 Inches</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
80	110	65	100	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	19	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	\pm	10 ppm
Oxygen	\pm	25 ppm
Nitrogen	\pm	25 ppm
Hydrogen	\pm	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176-M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
± 0.025 inch or $\pm 5\%$ whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material 6 inches wide or less and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II

DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width, Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.0129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken

transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by

longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection only the 0.020-inch diameter holes shall be used for calibration

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 0.1 hour

SPECIFICATION

SEAMLESS TUBING AND PIPE: D-43
(Cb-10W-1Zr-0.1C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: D-43
(Cb-10W-1Zr-0.1C) ALLOY

1. SCOPE

1.1. Scope. This specification covers D-43 (Cb-10W-1Zr-0.1C) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not

greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Carbon	1200	800	± 50
Oxygen	200	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	20	-	+ 5

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Carbon	1200	800	± 50

TABLE I
CHEMICAL COMPOSITION
D-43 (Cb-10W-1Zr-0.1C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	800	1200
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	100
Tantalum	-	1000
Molybdenum	-	200
Nickel	-	200
Cobalt	-	50
Iron	-	200
Tungsten	9.0 w/o	11.0 w/o
Zirconium	0.75 w/o	1.25 w/o
Columbium	Remainder	-

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Max.</u>	<u>Min.</u>	
Oxygen	400	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	20	-	+ 5

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>		<u>% R_x Minimum</u>
Less than 0.010	6	2		90
0.010 to 0.065	6	2		90
0.065 to 0.125	5	2		90
0.125 to 0.250	4	2		90
0.250 to 0.500	3	3		90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°+85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %(1)</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	
70	90	50	70	15

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2200	12	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the check analyses of analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 9).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall $\frac{3}{4}$ inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2 and 5.4.3 shall be mutually agreed upon by the vendor and the purchaser prior to the placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020 inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration

piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this

specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis either made or requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right- hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-222
(Ta-10.4W-2.4Hf-0.01C) ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

SEAMLESS TUBING AND PIPE: T-222
(Ta-10.4W-2.4Hf-0.01C) ALLOY

1. SCOPE

1.1. Scope. This specification covers T-222 (Ta-10.4W-2.4Hf-0.01C) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for
Designating Significant Places
in Specified Limiting Values

ASTM Designation E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the

purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Maximum</u>	<u>Minimum</u>	
Carbon	175	80	± 10
Oxygen	150	-	+ 20
Nitrogen	75	-	+ 10
Hydrogen	10	-	+ 2

For Wall Thicknesses Less Than 0.020 Inch

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Maximum</u>	<u>Minimum</u>	
Carbon	175	80	± 10
Oxygen	300	-	+ 20
Nitrogen	100	-	+ 10
Hydrogen	10	-	+ 2

TABLE I
CHEMICAL COMPOSITION
T-222 (Ta-10.4W-2.4Hf-0.01C) ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>
Carbon	80	175
Nitrogen	-	50
Oxygen	-	100
Hydrogen	-	10
Columbium	-	1000
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Vanadium	-	20
Tungsten	9.6 w/o	11.2 w/o
Hafnium	2.2 w/o	2.8 w/o
Tantalum	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Wall Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

<u>Ultimate Tensile Strength, ksi</u>		<u>0.2% Yield Strength, ksi</u>		<u>Elong., %(1)</u>
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
105	125	100	120	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2400	30	20

Chemical analyses of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of analytical results:

Carbon	±	10 ppm
Oxygen	±	25 ppm
Nitrogen	±	25 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

TABLE II
PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

<u>Nominal OD</u> <u>Inches</u>	<u>OD</u> <u>Inch</u>	<u>ID</u> <u>Inch</u>	<u>Wall</u> <u>Thickness</u> <u>%</u>
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agree upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding, provided the wall thickness is not decreased below that permitted in Table II (refer to page 8).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all test and inspection of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is

being furnished in accordance with this specification. The inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2. and 5.4.3. shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements.

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have an 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared

to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle

box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

APPENDIX B
REVISED SPECIFICATIONS FOR Cb-1Zr ALLOY

SPECIFICATION

SEAMLESS TUBING AND PIPE: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

- CONTINUED

DATE

29 Nov. 1965

NO.

01-0004-00-D

1. SCOPE

1.1. Scope. This specification covers columbium-1% zirconium alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of Metallic
Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Designating
Significant Places in Specified Limit-
ing Values

ASTM Designation (Pending)

Methods for Chemical Analysis of
Reactor and Commercial Columbium

ASTM Designation E112-61
(1961)

Estimating Average Grain Size of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspection

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. After the final reduction, the finished product shall be given a final vacuum anneal for one hour at a minimum temperature of 2200°F. The amount of total reduction from the turned ingot to the finished product shall exceed 75%; the amount of final reduction since the last recrystallization anneal and prior to the final recrystallization anneal shall exceed 30%.

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SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
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The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment, and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

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SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

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DATE

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NO.

01-0004-00-D

TABLE I
CHEMICAL COMPOSITION
COLUMBIUM-1% ZIRCONIUM ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>	<u>Analysis</u> <u>Requirement</u>
Carbon	50	200	↕ Each Ingot ↕
Nitrogen	--	100	
Oxygen	100	300	
Hydrogen	--	10	
Zirconium	0.80 w/o	1.2 w/o	
Iron	--	50	
Tantalum	--	1000	
Titanium	--	200	
Silicon	--	100	
Boron	--	2	↕ When So Specified ↕
Tungsten	--	200	
Molybdenum	--	200	
Cadmium	--	5	
Cobalt	--	30	
Lead	--	50	
Manganese	--	50	
Nickel	--	50	
Vanadium	--	50	
Hafnium	--	100	
Columbium, by Difference	98.5 w/o	--	

SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

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Element	Check Analysis Limits, ppm		Permissible Variations in Check Analysis, ppm
	Min.	Max.	
Carbon	50	200	+ 10
Oxygen	100	300	+ 20
Nitrogen	-	100	+ 10
Hydrogen	-	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

Product Wall Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	% R _x Minimum
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

For Wall Thickness 0.020 Inch or Greater

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elongation, % ⁽¹⁾
Minimum	Maximum	Minimum	Maximum	Minimum
35	75	20	60	20

For Wall Thicknesses Less Than 0.020 Inch

40	75	30	60	20
----	----	----	----	----

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Test Specimens.

SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

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3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3.):

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2000	10	15

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.7.5. Hardness Test. Rockwell hardness tests shall be made on the inside surfaces of specimens cut from tubes having wall thicknesses greater than 0.015 inch. The tubes shall have a Rockwell hardness number not exceeding B-90 in the heat treated condition.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 7).

SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

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TABLE II

PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD Inches	OD Inch	ID Inch	Wall Thickness %
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: -----

- (1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.
- (2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.
- (3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.
- (4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

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SEAMLESS TUBING AND PIPE: COLUMBIUM-1%
ZIRCONIUM ALLOY

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DATE

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3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 7).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Samples Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the

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29 November 1965
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SPECIFICATION

SEAMLESS TUBING AND PIPE: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SP 1973 A

SEAMLESS TUBING AND PIPE: Columbium-1%
Zirconium Alloy - CONTINUED

DATE

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NO.

01-0004-01-D

The following exceptions to specification No. 01-0004-00-D are applicable:

Paragraph 3.5.2. Final Product Composition - Minimum analyses requirement for carbon and oxygen content in Table I is deleted.

Paragraph 3.5.3. Check Analysis - Minimum limit requirement is deleted.

Paragraph 3.7.1. Room Temperature Tensile Properties - Minimum ultimate tensile strength and minimum 0.2% yield strength requirement are deleted.

Paragraph 3.7.2. Stress-Rupture Test requirement is deleted.

Paragraph 5.4.3. Stress-Rupture Test requirement is deleted.

Paragraph 5.7.1.1. Radiographic requirement is deleted.

All items shall clearly be identified with the specification number 01-0004-01-D.

SPECIFICATION

BAR AND ROD: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS.3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

BAR AND ROD: Columbium-1% Zirconium Alloy

- CONTINUED

DATE

29 Nov. 1965

NO.

01-0052-00-B

1. SCOPE

1.1. Scope. This specification covers columbium-1% zirconium alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

BAR AND ROD: Columbium-1% Zirconium Alloy

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DATE

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3.3. Processing. The starting stock size, processing temperatures percent-ages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. After the final reduction, the finished product shall be given a final vacuum anneal for one hour at a minimum temperature of 2200°F. The amount of total reduction from the turned ingot to the finished product shall exceed 75%; the amount of final reduction since the last recrystallization anneal and prior to the final recrystallization anneal shall exceed 30%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment, and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr.

3.4.2. Heat Treatment. All mill products to be annealed shall be thoroughly degreased, chemically cleaned, and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

BAR AND ROD: Columbium-1% Zirconium Alloy

- CONTINUED

DATE

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NO.

01-0052-00-B

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Minimum</u>	<u>Maximum</u>	
Carbon	50	200	± 10
Oxygen	100	300	± 20
Nitrogen	--	100	± 10
Hydrogen	--	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

<u>Product Diameter or Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

BAR AND ROD: Columbium-1% Zirconium Alloy

- CONTINUED

DATE

29 Nov. 1965

NO.

01-0052-00-B

TABLE I

CHEMICAL COMPOSITIONCOLUMBIUM-1% ZIRCONIUM ALLOY

<u>Element</u>	<u>Minimum Content</u> <u>ppm</u>	<u>Maximum Content</u> <u>ppm</u>	<u>Analysis</u> <u>Requirement</u>
Carbon	50	200	↕ Each Ingot ↕
Nitrogen	--	100	
Oxygen	100	300	
Hydrogen	--	10	
Zirconium	0.80 w/o	1.2% w/o	
Iron	--	50	
Tantalum	--	1000	
Titanium	--	200	
Silicon	--	100	
Boron	--	2	↕ When So Specified ↕
Tungsten	--	200	
Molybdenum	--	200	
Cadmium	--	5	
Cobalt	--	30	
Lead	--	50	
Manganese	--	50	
Nickel	--	50	
Vanadium	--	50	
Hafnium	--	100	
Columbium, by Difference	98.5 w/o	-	

BAR AND ROD: Columbium-1% Zirconium Alloy

- CONTINUED

DATE

29 Nov. 1965

NO.

01-0052-00-B

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., % in 4D
<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Minimum</u>
35	75	20	60	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3.).

<u>Test Temp., °F</u>	<u>Stress, ksi</u>	<u>Minimum Life Hours</u>
2000	10	5

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Hardness. The Rockwell Hardness of the product shall not exceed B-90 in the heat treated condition.

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

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3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25-inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled, square and rectangular shapes shall have the following tolerances:

<u>Thickness</u>	<u>Length</u>	<u>Width</u>
± 0.025 inch or $\pm 5\%$ whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

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TABLE IIPERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

<u>Diameter Inches</u>	<u>Diameter Variation Inch</u>	<u>Out-of-Roundness Inch</u>
0.125 to 0.281	+ 0.002, - 0.002	0.004
Over 0.281 to 0.406	+ 0.010, - 0.005	0.008
Over 0.406 to 0.625	+ 0.010, - 0.005	0.012
Over 0.625 to 0.875	+ 0.015, - 0.005	0.015
Over 0.875 to 1.000	+ 0.020, - 0.005	0.015
Over 1.000 to 1.375	+ 0.020, - 0.010	0.018
Over 1.375 to 1.500	+ 0.020, - 0.015	0.020
Over 1.500 to 1.625	+ 0.025, - 0.015	0.020
Over 1.625 to 2.000	+ 0.030, - 0.030	0.025
Over 2.000 to 2.500	+ 0.032, - 0.032	0.025
Over 2.500 to 3.250	+ 0.032, - 0.032	0.027
Over 3.250 to 3.500	+ 0.045, - 0.045	0.040

Centerless Ground Rounds

0.0625 to 2.0	+ 0.002, - 0.002
Over 2.0	+ 0.003, - 0.002

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4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., samples may be taken transverse to the final working direction from bar of sufficient width or from bar greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium".

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5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials". Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hardness Test - one per lot per ingot

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5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection". The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be

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made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of $1/2$ the thickness or diameter or, if the thickness exceeds 0.750 inch, $1/4$, $1/2$ and $3/4$ the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at $1/4$ and $1/2$ the thickness if the thickness exceeds 0.750-inch shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notches.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless

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otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

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TestChemical composition and di-
mensional tolerances (when
expressed decimally)

Tensile strength

Elongation

Rupture life

Rounded-Off Unit for
Observed or Calculated ValueNearest unit in the last right-
hand place of figures of the
specified limit

Nearest 100 psi

Nearest 1%

Nearest 0.1 hour

SPECIFICATION

BAR AND ROD: COLUMBIUM-1% ZIRCONIUM

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

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The following exceptions to specification No. 01-0052-00-B are applicable:

Paragraph 3.5.2. Final Product Composition - Minimum analyses requirement for carbon and oxygen content in Table I is deleted.

Paragraph 3.5.3. Check Analysis - Minimum limit requirement is deleted.

Paragraph 3.7.1. Room Temperature Tensile Properties - Minimum ultimate tensile strength and minimum 0.2% yield strength requirement are deleted.

Paragraph 3.7.2. Stress-Rupture requirement is deleted.

Paragraph 5.4.3. Stress-Rupture requirement is deleted.

Paragraph 5.7.1.1. Radiographic requirement is deleted.

All items shall clearly be identified with the specification number 01-0052-01-B.

SPECIFICATION

SHEET, PLATE, AND STRIP: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

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1. SCOPE

1.1. Scope. This specification covers columbium-1% zirconium alloy in sheet, plate, and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T
(26 December 1957)

Method of Tension Testing of
Metallic Materials

ASTM Designation E29-58T
(1958)

Recommended Practices for Des-
ignating Significant Places in
Specified Limiting Values

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

ASTM E112-61
(1961)

Estimating Average Grain Size
of Metals

AMS 2242A
(1 December 1950)

Tolerances, Corrosion and Heat
Resistant Sheet, Strip and Plate

AMS 2635
(15 August 1958)

Radiographic Inspection

AMS 2645
(1 March 1955)

Fluorescent Penetrant Inspec-
tion

AMS 2646
(1 March 1955)

Contrast Dye Penetrant Inspec-
tion

MAB-176-M
(6 September 1961)

Evaluation Test Methods for
Refractory Metal Sheet Mater-
ials

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3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. After the final reduction, the finished product shall be given a final vacuum anneal for one hour at a minimum temperature of 2200°F. The amount of total reduction from the turned ingot to the finished product shall exceed 75%; the amount of final reduction since the last recrystallization anneal and prior to the final recrystallization anneal shall exceed 30%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment, and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination.

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caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, ppm</u>		<u>Permissible Variations in Check Analysis, ppm</u>
	<u>Min.</u>	<u>Max.</u>	
Carbon	50	200	± 10
Oxygen	100	300	± 20
Nitrogen	--	100	± 10
Hydrogen	--	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

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TABLE I

CHEMICAL COMPOSITIONCOLUMBIUM-1% ZIRCONIUM ALLOY

<u>Element</u>	<u>Minimum Content</u> ppm	<u>Maximum Content</u> ppm	<u>Analysis</u> <u>Requirement</u>
Carbon	50	200	 Each Ingot 
Nitrogen	--	100	
Oxygen	100	300	
Hydrogen	--	10	
Zirconium	0.80 w/o	1.2 w/o	
Iron	--	50	
Tantalum	--	1000	
Titanium	--	200	
Silicon	--	100	
Boron	--	2	 When So Specified 
Tungsten	--	200	
Molybdenum	--	200	
Cadmium	--	5	
Cobalt	--	30	
Lead	--	50	
Manganese	--	50	
Nickel	--	50	
Vanadium	--	50	
Hafnium	--	100	
Columbium, by difference	98.5 w/o	-	

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Zirconium Alloy			
<u>Product Thickness, Inches</u>	<u>Minimum Allowable ASTM Grain Size No.</u>	<u>Allowable Spread in ASTM Grain Size Nos. in Any One Item</u>	<u>% R_x Minimum</u>
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F):

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., % in 2 Inches
Minimum	Maximum	Minimum	Maximum	Minimum
For sheet less than 0.187" thick				
40	75	30	60	20
For plate 0.187" thick or greater				
35	75	25	60	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3.).

Test Temp., °F	Stress, ksi	Minimum Life Hours
2000	10	5

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results.

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Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.7.4. Hardness. Rockwell hardness tests shall be made on all products greater than 0.015 inch thick; the Rockwell hardness number shall not exceed B-90 in the heat treated condition.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

<u>Thickness</u>	<u>Width</u>	<u>Length</u>
± 0.025 inch or ± 5% whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerances on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

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3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material less than 6 inches wide and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \% \text{ Flatness Deviation}$$

where:

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

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TABLE IIDIMENSIONAL TOLERANCES FOR SHEET AND STRIP

<u>Material Thickness, Inch</u>	<u>Width Inches</u>	<u>Thickness Tolerances, Inch</u>
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

<u>Material Thickness, Inch</u>	<u>Width Tolerances, Inch</u>
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

<u>Material Thickness, Inch</u>	<u>Length Tolerances, Inch</u>
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

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4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.50 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet

SHEET, PLATE, AND STRIP: Columbium-1%
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and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1×10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

SHEET, PLATE, AND STRIP: Columbium-1%
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Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hardness Test - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

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Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far size or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving and 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection, or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750-inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection, only the 0.020-inch diameter holes shall be used for calibration.

SHEET, PLATE, AND STRIP: Columbium-1%
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5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

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Zirconium Alloy

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5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITION

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

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7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

<u>Test</u>	<u>Rounded-Off Unit for Observed or Calculated Value</u>
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

SHEET, PLATE, AND STRIP: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

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SHEET, PLATE, AND STRIP: Columbium-1%
Zirconium Alloy . CONTINUED

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The following exceptions to specification No. 01-0053-00-B are applicable:

Paragraph 3.5.2. Final Product Composition - Minimum analyses requirements for carbon and oxygen contents in Table I are deleted.

Paragraph 3.5.3. Check Analysis - Minimum limit requirement is deleted.

Paragraph 3.7.1. Room Temperature Tensile Properties - Minimum ultimate tensile strength and minimum 0.2% yield strength requirements are deleted.

Paragraph 3.7.2. Stress-Rupture requirement is deleted.

Paragraph 5.4.3. Stress-Rupture requirement is deleted.

Paragraph 5.7.1.1. Radiographic requirement is deleted.

All items shall clearly be identified with the specification number 01-0053-01-B.

SPECIFICATION

FOIL; COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

FOIL: Columbium-1% Zirconium Alloy - CONTINUED

DATE

10 June 1965

NO.

01-0054-00-A

1. SCOPE

1.1. Scope. This specification covers columbium-1% zirconium alloy in foil form intended for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis of
Reactor and Commercial Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition through the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

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DATE

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3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definition. Foil includes material less than 12 inches wide and up to including 0.010-inch thick.

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

<u>Materials Thickness Inches</u>	<u>Thickness Tolerances Inch</u>	<u>Width Tolerance Inch</u>
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	+0.001	+0.031, -0.000
0.005 to 0.010	+0.0015	+0.031, -0.000

FOIL: Columbium-1% Zirconium Alloy . CONTINUED

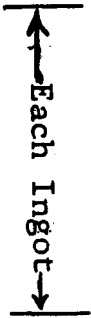
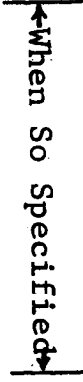
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TABLE ICHEMICAL COMPOSITIONCOLUMBIUM-1% ZIRCONIUM ALLOY

<u>Element</u>	<u>Maximum Content</u> <u>ppm</u>	<u>Analysis</u> <u>Requirements</u>
Carbon	150	 Each Ingot
Nitrogen	100	
Oxygen	300	
Hydrogen	10	
Zirconium	0.8 - 1.2% (range)	
Iron	50	
Tantalum	1000	
Titanium	200	
Silicon	100	
Boron	2	 When So Specified
Tungsten	200	
Molybdenum	200	
Cadmium	5	
Cobalt	50	
Lead	50	
Manganese	50	
Nickel	50	
Vanadium	50	
Hafnium	100	
Columbium, by difference	98.5% min.	

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FOIL: Columbium-1% Zirconium Alloy - CONTINUED	10 June 1965	01-0054-00-A

3.8. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium".

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

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FOIL: Columbium-1% Zirconium Alloy - CONTINUED	DATE 10 June 1965	NO. 01-0054-000-A
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4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITION

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

SPECIFICATION

WIRE: COLUMBIUM-1% ZIRCONIUM ALLOY

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

WIRE: Columbium-1% Zirconium Alloy - CONTINUED

DATE

10 June 1965

NO.

01-0055-00-A

1. SCOPE

1.1. Scope. This specification covers columbium-1% zirconium alloy in wire for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)

Methods for Chemical Analysis
of Reactor and Commercial
Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

WIRE: Columbium-1% Zirconium Alloy

- CONTINUED

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3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Material Greater than 0.030 Inch in Diameter

<u>Element</u>	<u>Check Analysis Limits, Max., ppm</u>	<u>Permissible Variations in Check Analysis, ppm</u>
Carbon	100	+ 10
Oxygen	250	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

For Material 0.030 Inch and Less in Diameter

Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

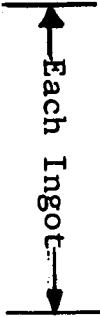
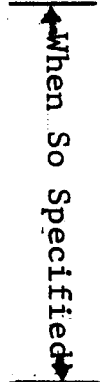
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WIRE: Columbium-1% Zirconium Alloy - CONTINUED

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TABLE ICHEMICAL COMPOSITIONCOLUMBIUM-1% ZIRCONIUM ALLOY

<u>Element</u>	<u>Maximum Content</u> <u>ppm</u>	<u>Analysis</u> <u>Requirements</u>
Carbon	100	 Each Ingot
Nitrogen	100	
Oxygen	250	
Hydrogen	10	
Zirconium	0.8 - 1.2% (range)	
Iron	50	
Tantalum	1000	
Titanium	200	
Silicon	100	 When So Specified
Boron	2	
Tungsten	200	
Molybdenum	200	
Cadmium	5	
Cobalt	30	
Lead	50	
Manganese	50	
Nickel	50	
Vanadium	50	
Hafnium	100	
Columbium, by difference	98.5% min.	

WIRE: Columbium-1% Zirconium Alloy - CONTINUED

DATE

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NO.

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3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

<u>Diameter, Inch</u>	<u>Diameter Variation, Inch</u>
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

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WIRE: Columbium-1% Zirconium Alloy - CONTINUED

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01-0055-00-A

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

APPENDIX C
QUALITY ASSURANCE MATERIAL CONTROL RECORD FORMS

SP 1068
1070
1069

Contract NAS - _____

[illegible]

RAW MATERIAL TEST RECORD

M. C. N. _____

Chem. Analysis _____ By _____

Hardness _____ By _____

Grain Size _____ By _____

Stress-Rupture _____ By _____

Tensile _____ By _____

Ultrasonic _____ By _____

X-Ray _____ By _____

Zyglo _____ By _____

Flare _____ By _____

Hydrostatic _____ By _____

Eddy Current _____ By _____

Visual _____ By _____

_____ By _____

_____ By _____

_____ By _____

Remarks _____

Disposition: Acc. _____ By _____

Rej. _____ By _____

SP 1070

[illegible]

SP 1069

APPENDIX D
PROCESS SPECIFICATIONS

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SPPS-3B
24 September 1963
Page 1 of 9

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

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SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for fusion welding the columbium-1% zirconium alloy by the inert-gas shielded tungsten arc-welding process. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents

ASTM Designation B297-55T
(1 February 1955)

Tungsten Arc-Welding Electrodes

AMS 2635
(15 August 1955)

Radiographic Inspection

3. REQUIREMENTS

3.1. Materials

3.1.1. Inert gases used shall be Bureau of Mines Grade A helium or argon, welding grade, 99.99% minimum purity, -80°F maximum dew point. The combined total of oxygen and water vapor shall be less than 0.005% by volume.

3.1.2. Tungsten electrodes, class EWTH-2, shall conform to ASTM Designation B297-55T, "Tungsten Arc-Welding Electrodes".

3.1.3. Welding filler material composition shall conform to the specification for the base metal.

3.2. Equipment

3.2.1. A direct current, arc-welding machine, equipped with a contactor and a foot-operated remote welding-current control, shall be used.

3.2.2. The welding shall be done in an enclosed chamber that can be evacuated to less than 1×10^{-4} torr. The leak rate shall result in a pressure increase of not more than 15 microns/hour starting at 1×10^{-3} torr pressure. The chamber shall be equipped with glove ports and neoprene rubber gloves that are sealed gas-tight to the ports.

3.2.3. The chamber shall be equipped with a water-cooled tungsten arc-welding torch. The water-cooling passage of the torch shall be permanently sealed. The water-cooled cable and hose connections shall be leak tight. The inert-gas shield line shall not be used.

3.3. Cleaning

3.3.1. The parts to be joined shall be free of dirt, grease, cutting compounds, or any extraneous material which will attack or react with the metal during welding or contaminate the inert-gas atmosphere. The metal adjacent to the weld shall be chemically cleaned to remove surface oxide and/or nitride with a solution of 20%HF-20%HNO₃-60%H₂O by volume.

3.3.2. The welding filler-wire shall be washed with acetone and shall be chemically cleaned to remove dirt, oxide, grease, drawing compounds using the above solution.

3.4. Fixtures

3.4.1. Design. The fixtures used to position the parts shall hold the parts in proper alignment and minimize distortion.

3.4.2. Materials. The fixtures shall be made of materials that do not corrode or rust in air. To avoid metallic contamination, the fixture components that contact the columbium parts near the weld joint shall be made of molybdenum, tungsten, or columbium.

3.4.3. Cleaning. The fixture shall be clean and free of surface contamination.

3.5. Welding Procedures

3.5.1. Welding of components shall be done using equipment and procedures which conform to Section 4. The parts to be welded and the required fixturing shall be loaded into the welding chamber, evacuated of air to

1×10^{-4} torr or lower. The chamber shall then be filled with inert-gas conforming to paragraph 3.1. Before removing the glove port cover plates, the evacuation line to the glove ports shall be closed.

3.5.2. Direct current, straight-polarity power shall be used for welding. The welding torch shall be used without a gas cup and without gas flow.

3.5.3. Before welding the columbium parts, the purity of the welding atmosphere shall be checked by making fusion weld beads on titanium sheet or bar. If the titanium weld shows any discoloration, the inert atmosphere shall be considered unsuitable for welding columbium alloys.

3.5.4. Precautions shall be taken during welding to avoid contamination of the weld metal by the tungsten electrode.

3.5.5. The completed weldment shall be left in the chamber until it cools to below 400°F.

3.5.6. In repair welding, the weld defects shall be removed by machining or filing. Unless approved by the General Electric project engineer, grinding is prohibited. The area shall then be rewelded, according to the requirements of this specification, and reinspected.

3.6. Postweld Heat Treatment

3.6.1 The weld and adjacent heat-affected zone shall be postweld annealed at 2200°F, plus or minus 25°F, for one hour in a vacuum of 1×10^{-4} torr or lower. The annealing may be local at the weld joint or the entire weldment may be annealed in a vacuum furnace. The procedures and equipment used shall conform to paragraph 4.3.

3.6.2. Localized heating shall be done using a tantalum or tungsten heater. Dense vitrified alumina (96% purity) shall be used as the heater electrical insulation and shall not contact the columbium alloy during heat treatment. The temperature of the weld joint shall be measured by a Pt/Pt-Rh thermocouple that is resistance spot-tacked to the weld metal. After heat treatment the thermocouple spot-tack shall be filed off.

3.7. Field Welding

3.7.1 Field welding is welding of joints in components which cannot be done in vacuum purged chambers because of size and space limitations.

3.7.2. Field welding shall be done using an inert-gas purged enclosure or auxiliary inert-gas shield devices around the joint to displace the air from the weld. Wherever possible, chill blocks shall be used to remove heat from the weld area as rapidly as possible. (The chills may be a part of the gas shield device.)

3.7.3. The welding shall be done using an inert-arc welding torch with a ceramic torch gas shield cup. The torch inert-gas shield shall be used.

3.7.4. The welding process and inert-gas shielding equipment shall be qualified as in Section 4.

3.8. Defect Limits. The completed weldment shall be examined for defects visually and by ultrasonic or x-ray techniques, or both. Welds which do not meet the following quality requirements of this specification are unacceptable.

3.8.1. Visual and Radiographic. Welds shall be reasonably smooth and uniform in appearance and free of the following defects by visual and radiographic inspection:

1. Cracks in the weld and adjacent base metal,
2. Crater checks or cracks,
3. Surface inclusions and pores,
4. Cold layers in deposited weld metal,
5. Overlap of weld metal on base metal (not fused),
6. Undercutting of the base metal,
7. Depression of the weld below the base metal,
8. Unfilled weld craters or stops,
9. Damage to the weld and adjacent base metal by contamination from the shielding gas or from foreign materials or metals,
10. Lack of fusion of the weld deposit to the base metal,
11. Incomplete fusion and penetration to the root of the joint (back side of butt and groove welds),
12. Porosity in the weld metal,
13. Tungsten inclusions having
 - a. the longest dimension greater than 0.010 inch in welds in material up to 0.10 inch thick and greater than 10% of the metal thickness in material 0.10 inch thick and above
 - b. spacing between two adjacent tungsten inclusions less than three times the metal thickness
 - c. more than three inclusions per inch of weld length.

3.8.2. Ultrasonic. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication. For tubing, inside diameter (or under side) defects shall be compared to the indication from the notch on the inside diameter (or under side), and outside diameter (or upper side) defects shall be compared to the indication from the notch on the outside diameter (or upper side). Indications less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the upper side (or outside diameter) calibration indication. Defects more than half the distance from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter (or under surface) notch.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by x-ray or ultrasonic techniques, or both.

4.1.1. X-ray Inspection

4.1.1.1. X-ray inspection shall be done according to AMS 2635, "Radiographic Inspection". Fine grain film shall be used for maximum sensitivity. The penetrameter thickness and hole sizes shall be based on the smaller section thickness being joined. The radiographic technique shall be considered satisfactory if the smallest hole in the penetrameter can be clearly distinguished on the radiograph.

4.1.1.2. The radiographs shall be taken in positions that will best delineate lack of weld penetration and fusion. Wherever possible, the film shall be placed on the back side of the weld joint.

4.1.1.3. When the back side of the weld is not accessible and the radiation must pass through a double thickness, only that portion of the weld next to the film shall be considered.

4.1.1.4. Radiographic interpretation for weld quality shall be done by the General Electric project engineer or his representative.

4.1.2. Ultrasonic Inspection

4.1.2.1. When required by the drawing, welded joints shall be ultrasonically rather than radiographically inspected..

4.1.2.2. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Shear wave technique

shall be used with the beam traverse perpendicular to the weld centerline. Inspection shall be from both directions toward the weld. For welds in tubing, a transducer focused to the diameter of the tubing shall be used with a maximum axial length on the transducer of 0.5 inch. For welds in plate, a spherically focused transducer or a cylindrically focused transducer of less than 1.5 inch diameter focus and 0.5 inch or less long shall be used with its axis parallel to the weld centerline.

4.1.2.3. Calibration shall be on notches cut in a tube or plate of similar material, preferably near a weld of similar nature. These circumferential notches shall be: depth, 3% of the original wall or plate thickness; width, not more than the depth; length, at least one beam width. One notch shall be placed on the inside diameter or under side near the weld; the other notch shall be placed on the outside diameter or upper side near the weld but on the opposite side of the weld or at least two beam widths away (at the same distance from the weld) if on the same side of the weld. Focusing shall be done to maximize the indication from the notch on the under side or inside diameter. After focusing is completed, whichever indication is larger shall be set at 80% and the amplitude of the indication from the other notch shall be recorded. Distance corresponding to the wall or plate thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. All testing since previous calibration shall be repeated whenever the calibration gain changes by more than 10%.

4.2. Welding Equipment and Procedure

4.2.1. Before welding actual columbium alloy parts and components, the welding equipment and procedures shall be qualified by welding columbium - 1% zirconium alloy and testing as follows.

4.2.2. A butt-welded joint will be made in the vacuum purged welding chamber in the columbium - 1% zirconium alloy. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch.

4.2.3. Four strips shall be cut from the welded joint with the weld running transverse to the length. The minimum width of three strips shall be four times the base metal thickness or 1/2 inch, whichever is smaller. The welded strips shall be annealed at 2200°F for one hour in vacuum.

4.2.4. Three welded strips shall be bend tested using a 75-degree V-block and mating V-punch with a radius three times the base metal thickness. Each weld is to be placed face down and centered under the punch radius. The reinforcement on the back side shall be removed before testing.

4.2.5. All three test strips shall withstand a transverse bend through 90 degrees without cracking. The weld and heat-affected zones shall be visually examined for cracks at a magnification of 10X. All three specimens must pass the bend test requirements.

4.2.6. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the fourth strip. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. The impurity content shall not exceed the following:

<u>Element</u>	<u>Amount (ppm)</u>
Oxygen	500
Nitrogen	350
Carbon	150
Hydrogen	15

4.3. Postweld Annealing Procedures and Equipment

4.3.1. Before annealing welded joints in components, the annealing procedures and equipment shall be qualified as described to insure that the weldments are not contaminated and embrittled during the operation.

4.3.2. A sample welded joint simulating the actual weldment shall be welded using qualified equipment and procedures. The weld shall be heat-treated at 2200°F, plus or minus 25°F, for one hour in a vacuum using the procedures and equipment that will be used for production welds. If localized heat-treating equipment is used, the temperature of the weld shall be measured by a Pt/Pt-Rh thermocouple which is resistance spot-tacked to the part.

4.3.3. Three strips shall be cut from the joint. Two shall have a width four times the base metal thickness but not less than 1/2 inch. The third one shall be sufficiently wide to obtain a weld metal, chemical analysis sample. The weld shall run transverse to the length of the strip at midpoint.

4.3.4. One transverse weld bend strip shall be bend tested as annealed, and the other shall be bend tested after aging at 1500°F for fifty hours in a vacuum furnace at a pressure of 1×10^{-5} torr maximum. The bend test procedures shall be those described in paragraph 4.2. Both bend strips must pass the test requirements.

4.3.5. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen and carbon shall be made. The impurity content shall not exceed the following:

<u>Element</u>	<u>Amount (ppm)</u>
Oxygen	500
Nitrogen	350
Carbon	150
Hydrogen	15

4.3.6. Qualifying both welding and annealing procedures and equipment simultaneously is permissible. The test results must meet the requirements of paragraphs 4.2. and 4.3.

4.4. Reports. A report shall be submitted with the finished parts and shall include all vacuum, temperature, and inert gas purity measurements applicable to welding and heating, the X-rays of the weld joints, and/or when required, location and approximate depth of ultrasonic indications found. Ultrasonic reports shall also include the ultrasonic equipment and transducer used and calibration data. The above reports and all accompanying X-rays, charts, etc., shall reference the drawing number, part number, and serial number of each part represented and, when multiple welds on a part are made in separate environments, the particular weld to which the information is applicable.

Note: These reports shall be retained by quality assurance, should subsequent weld evaluation be desired.

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SPPS-3C
24 September 1963
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SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for fusion welding the columbium-1% zirconium alloy by the inert-gas shielded tungsten arc-welding process. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents.

ASTM Designation B297-55T
(1 February 1955)

Tungsten Arc-Welding
Electrodes

AMS 2635
(15 August 1955)

Radiographic Inspection

3. REQUIREMENTS

3.1. Materials

3.1.1. Inert gases used shall be Bureau of Mines Grade A helium or argon, welding grade, 99.99% minimum purity, -80°F maximum dewpoint. The inert-gases shall be purified to contain less than 1 ppm active impurities by volume.

3.1.2. Tungsten electrodes, class EWTH-2, shall conform to ASTM Designation B297-55T, "Tungsten Arc-Welding Electrodes."

3.1.3. Welding filler material composition shall conform to the specification for the base metal.

3.2. Equipment

3.2.1. A direct current, arc-welding machine, equipped with a contactor and a foot-operated remote welding-current control, shall be used.

3.2.2. The welding shall be done in an enclosed chamber that can be evacuated to less than 1×10^{-5} torr. The leak rate shall result in a pressure increase of not more than 5 microns/hour starting at 1×10^{-3} torr pressure. The chamber shall be equipped with glove ports and neoprene rubber gloves that are sealed gas-tight to the ports.

3.2.3. The chamber shall be equipped with a water-cooled tungsten arc-welding torch. The water-cooling passage of the torch shall be permanently sealed. The water-cooled cable and hose connections shall be leak tight. The inert-gas shield line shall not be used.

3.3. Cleaning

3.3.1. The parts to be joined shall be free of dirt, grease, cutting compounds, or any extraneous material which will attack or react with the metal during welding or contaminate the inert-gas atmosphere. The metal adjacent to the weld shall be chemically cleaned to remove surface oxide and/or nitride with a solution of 20%Hf-20%HNO₃-60%H₂O by volume.

3.3.2. The welding filler-wire shall be washed with acetone and shall be chemically cleaned to remove dirt, oxide, grease, drawing compounds using the above solution.

3.4. Fixtures

3.4.1. Design. The fixtures used to position the parts shall hold the parts in proper alignment and minimize distortion.

3.4.2. Materials. The fixtures shall be made of materials that do not corrode or rust in air. To avoid metallic contamination, the fixture components that contact the columbium parts near the weld joint shall be made of molybdenum, tungsten, or columbium.

3.4.3. Cleaning. The fixture shall be clean and free of surface contamination.

3.5. Welding Procedures

3.5.1. Welding of components shall be done using equipment and procedures which conform to Section 4. The parts to be welded and the required

fixturing shall be loaded into the welding chamber, evacuated of air to 1×10^{-5} torr or better. The chamber shall then be filled with inert-gas containing less than 1 ppm active impurities by volume, evacuated to less than 1×10^{-5} torr, and refilled with inert gas. Before removing the glove port cover plates, the evacuation line to the glove ports shall be closed.

3.5.2. Direct current, straight-polarity power shall be used for welding. The welding torch shall be used without a gas cup and without gas flow.

3.5.3. Before welding the columbium parts, the purity of the welding atmosphere shall be checked by making fusion weld beads on titanium sheet or bar. If the titanium weld shows any discoloration, the inert atmosphere shall be considered unsuitable for welding columbium alloys.

3.5.4. Prior to the first piece welded and subsequent to the last piece welded in each inert gas environment, weld bend specimens shall be prepared using the same filler wire material used to weld the intervening pieces. Each specimen shall be from 0.04 to 0.08 inch thick, 1 inch wide (minimum), and 2 inches long (minimum), with a longitudinal weld bead weighing 2.5 grams (minimum).

The above specimens and 10-gram (minimum) samples taken from each heat of sheet and filler wire used to make the bend specimens shall be submitted with the finished parts. Specimens and samples shall be tagged with the drawing number, part number, and serial number of each part represented by the samples and specimens, and, when multiple welds on a part are made in separate environments, shall describe the particular welds for which the specimens and samples are representative.

Note: These specimens and samples shall be retained by quality assurance for subsequent chemical analysis should an investigation of welding contamination be desired.

3.5.5. Precautions shall be taken during welding to avoid contamination of the weld metal by the tungsten electrode.

3.5.6. The completed weldment shall be left in the chamber until it cools to below 400°F.

3.5.7. In repair welding, the weld defects shall be removed by machining or filing. Unless approved by the General Electric project engineer, grinding is prohibited. The area shall then be rewelded, according to the requirements of this specification, and reinspected.

3.6. Postweld Heat Treatment

3.6.1. The weld and adjacent heat-affected zone shall be postweld annealed at 2200°F, plus or minus 25°F, for one hour in a vacuum of 1×10^{-5} torr

or better. The annealing may be local at the weld joint or the entire weldment may be annealed in a vacuum furnace. The procedures and equipment used shall conform to paragraph 4.3.

3.6.2. Localized heating shall be done using a heater having only refractory metals in the hot zone. Dense vitrified alumina (99.2% purity) shall be used as the heater electrical insulation in the cold zone of furnace. The temperature of the weld joint shall be measured by a Pt/Pt-Rh thermocouple that is resistance spot-tacked to the weld metal. After heat treatment the thermocouple spot-tack shall be filed off.

3.6.3. A columbium - 1% zirconium control specimen, weighing 5 grams or more, shall accompany the welded components or parts through postweld vacuum heat treatment.

The above specimens and a similar, but unheated, specimen of the control material shall be submitted with the finished parts. Specimens shall be tagged with the drawing number, part number, and serial number of each part represented by the specimens.

Note: These specimens shall be retained by quality assurance for subsequent chemical analysis, should an investigation of vacuum environment contamination be desired.

3.7. Defect Limits. The completed weldment shall be examined for defects visually and by ultrasonic or x-ray techniques, or both. Welds which do not meet the following quality requirements of this specification are unacceptable.

3.7.1. Visual and Radiographic. Welds shall be reasonably smooth and uniform in appearance and free of the following defects by visual and radiographic inspection:

1. Cracks in the weld and adjacent base metal,
2. Crater checks or cracks,
3. Surface inclusions and pores,
4. Cold layers in deposited weld metal,
5. Overlap of weld metal on base metal (not fused),
6. Undercutting of the base metal,
7. Depression of the weld below the base metal,
8. Unfilled weld craters or stops,
9. Damage to the weld and adjacent base metal by contamination from the shielding gas or from foreign materials or metals,
10. Lack of fusion of the weld deposit to the base metal,
11. Incomplete fusion and penetration to the root of the joint (back side of butt and groove welds),
12. Porosity in the weld metal,

13. Tungsten inclusions having
- a. the longest dimension greater than 0.010 inch in welds in material up to 0.10 inch thick and greater than 10% of the metal thickness in material 0.10 inch thick and above
 - b. spacing between two adjacent tungsten inclusions less than three times the metal thickness
 - c. more than three inclusions per inch of weld length.

3.7.2. Ultrasonic. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication. For tubing, inside diameter (or under side) defects shall be compared to the indication from the notch on the inside diameter (or under side), and outside diameter (or upper side) defects shall be compared to the indication from the notch on the outside diameter (or upper side). Indications less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the upper side (or outside diameter) calibration indication. Defects more than half the distance from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter (or under surface) notch.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by X-ray or ultrasonic techniques, or both.

4.1.1. X-ray Inspection

4.1.1.1. X-ray inspection shall be done according to AMS-2635, "Radiographic Inspection", Fine grain film shall be used for maximum sensitivity. The penetrameter thickness and hole sizes shall be based on the smaller section thickness being joined. The radiographic technique shall be considered satisfactory if the smallest hole in the penetrameter can be clearly distinguished on the radiograph.

4.1.1.2. The radiographs shall be taken in positions that will best delineate lack of weld penetration and fusion. Wherever possible, the film shall be placed on the back side of the weld joint.

4.1.1.3. When the back side of the weld is not accessible and the radiation must pass through a double thickness, only that portion of the weld next to the film shall be considered.

4.1.1.4. Radiographic interpretation for weld quality shall be done by the General Electric project engineer or his representative.

4.1.2. Ultrasonic Inspection

4.1.2.1. When required by the drawing, welded joints shall be ultrasonically rather than radiographically inspected.

4.1.2.2. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Shear wave technique shall be used with the beam traverse perpendicular to the weld centerline. Inspection shall be from both directions toward the weld. For welds in tubing, a transducer focused to the diameter of the tubing shall be used with a maximum axial length on the transducer of 0.5 inch. For welds in plate, a spherically focused transducer or a cylindrically focused transducer of less than 1.5 inch diameter focus and 0.5 inch or less long shall be used with its axis parallel to the weld centerline.

4.1.2.3. Calibration shall be on notches cut in a tube or plate of similar material, preferably near a weld of similar nature. These circumferential notches shall be: depth, 3% of the original wall or plate thickness; width, not more than the depth; length, at least one beam width. One notch shall be placed on the inside diameter or under side near the weld; the other notch shall be placed on the outside diameter or upper side near the weld but on the opposite side of the weld or at least two beam widths away (at the same distance from the weld) if on the same side of the weld. Focusing shall be done to maximize the indication from the notch on the under side or inside diameter. After focusing is completed, whichever indication is larger shall be set at 80% and the amplitude of the indication from the other notch shall be recorded. Distance corresponding to the wall or plate thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. All testing since previous calibration shall be repeated whenever the calibration gain changes by more than 10%.

4.2. Welding Equipment and Procedure

4.2.1. Before welding actual columbium alloy parts and components, the welding equipment and procedures shall be qualified by welding columbium - 1% zirconium alloy and testing as follows.

4.2.2. A butt-welded joint will be made in the vacuum purged welding chamber in the columbium - 1% zirconium alloy. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch. Welding filler material shall be sheared from the parent alloy.

4.2.3. Four strips shall be cut from the welded joint with the weld running transverse to the length. The minimum width of three strips shall be four times the base metal thickness or 1/2 inch, whichever is smaller.

4.2.4. Three welded strips shall be bend tested using a 75-degree V-block and mating V-punch with a radius three times the base metal thickness. Each weld is to be placed face down and centered under the punch radius. The reinforcement on the back side shall be removed before testing.

4.2.5. All three test strips shall withstand a transverse bend through 90 degrees without cracking. The weld and heat-affected zones shall be visually examined for cracks at a magnification of 10X. All three specimens must pass the bend test requirements.

4.2.6. Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the fourth strip. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. These chemical analyses shall demonstrate that the degree of environmental contamination did not exceed the following limits: increase in oxygen content--less than 50 ppm; increase in nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results.

Carbon	±	10 ppm
Oxygen	±	30 ppm
Nitrogen	±	30 ppm
Hydrogen	±	2 ppm

4.3. Postweld Annealing Procedures and Equipment

4.3.1. Before annealing welded joints in components, the annealing procedures and equipment shall be qualified as described to insure that the weldments are not contaminated and embrittled during the operation.

4.3.2. A sample welded joint simulating the actual weldment shall be welded using qualified equipment and procedures. The weld shall be heat-treated at 2200°F, plus or minus 25°F, for one hour in a vacuum using the procedures and equipment that will be used for production welds. If localized heat-treating equipment is used, the temperature of the weld shall be measured by a Pt/Pt-Rh thermocouple which is resistance spot-tack to the part.

4.3.3. Three strips shall be cut from the joint. Two shall have a width four times the base metal thickness but not less than 1/2 inch. The third one shall be sufficiently wide to obtain a weld metal, chemical analysis sample. The weld shall run transverse to the length of the strip at midpoint.

4.3.4. One transverse weld bend strip shall be bend tested as annealed, and the other shall be bend tested after aging at 1500°F for fifty hours in a vacuum furnace at a pressure of 1×10^{-5} torr maximum. The bend test procedures shall be those described in paragraph 4.2. Both bend strips must pass the test requirements.

4.3.5. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the third strip. These analyses shall demonstrate that the degree of environmental contamination did not exceed the limits set forth in paragraph 4.2.

4.3.6. Qualifying both welding and annealing procedures and equipment simultaneously is permissible. The test results must meet the requirements of paragraphs 4.2 and 4.3.

4.4. Reports. A report shall be submitted with the finished parts and shall include all vacuum, temperature, and inert gas purity measurements applicable to welding and heating, the X-rays of the weld joints, and/or when required, location and approximate depth of ultrasonic indications found. Ultrasonic reports shall also include the ultrasonic equipment and transducer used and calibration data. The above reports and all accompanying X-rays, charts, etc., shall reference the drawing number, part number, and serial number of each part represented and, when multiple welds on a part are made in separate environments, the particular weld to which the information is applicable.

Note: These reports shall be retained by quality assurance, should subsequent weld evaluation be desired.

SPECIFICATION

VACUUM BRAZING BIMETALLIC TUBE JOINTS

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

VACUUM BRAZING BIMETALLIC TUBE JOINTS

1. SCOPE

1.1. This specification establishes the requirements and outlines the procedure for vacuum brazing tube joints between refractory alloys (Mo-0.5Ti, Mo-TZM, Cb-1Zr) and Type 316 stainless steel or L-605 alloy.

1.2. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements included on the drawing supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

SPPS-10
(3 June 1963)

Operator Certification for High
Temperature, Vacuum Furnace Brazing
of Bimetallic Tube Joints

FPD B50T56-S1
(23 April 1958)

Cobalt Base Alloy Powder

AMS 2635
(15 August 1958)

Radiographic Inspection

3. MATERIALS

3.1. The brazing alloy shall conform to FPD specification B50T56-S1.

3.2. The binder material shall be Nicrobrazecement or its equivalent.

3.3. The stop-off material shall be Nicrobrazegreen or its equivalent.

3.4. The cleaning agent shall be acetone.

4. REQUIREMENTS

4.1. Equipment

4.1.1. The vacuum furnace shall be capable of producing a vacuum at least 1×10^{-4} torr at 2200°F. The vertical, uniform hot zone shall have a 6-inch minimum length and a 3-inch minimum diameter. The leak rate, starting at 1×10^{-3} torr, shall result in a pressure increase not exceeding 2 microns/hour per cubic foot of chamber.

4.1.2. Auxiliary equipment shall be capable of monitoring furnace vacuum and part temperature (plus or minus 5°F).

4.2. Furnace Operator Certification. Only those operators certified in accordance with specification SPPS-10 shall perform brazing.

4.3. Cleaning. All components of the bimetallic joint shall be cleaned by one of the methods given in paragraph 5.1.

4.4. Joint Fit-Up. Joint fit-up shall be as specified on the engineering drawing.

4.5. Braze Characteristics

4.5.1. The brazing alloy shall flow through the joint uniformly, forming smooth, even fillets.

4.5.2. Brazing alloy flow shall be limited to a 0.25-inch fillet width.

4.5.3. Brazements shall be free from cracks and other defects outlined in paragraph 6.

5. PROCEDURE

5.1. Cleaning

5.1.1. Molybdenum and Molybdenum Alloys

5.1.1.1. Parts shall be degreased with acetone or equivalent.

5.1.1.2. Parts shall be cleaned electrolytically and polished by immersion in a solution of 66% H_2SO_4 - 34% H_2O by volume maintained at room temperature. Current densities of 100 to 300 amp/ft² shall be used with the molybdenum as the anode. The blue oxide film that is formed shall be removed by alternate rinsing in distilled water and acetone. A minimum one-inch length from the joint end of the tube shall be cleaned.

5.1.1.3. Immediately after cleaning, the parts shall be stored in sealed polyethylene bags until they are prepared for brazing.

5.1.2. Columbium and Columbium Alloys

5.1.2.1. Parts shall be degreased with acetone or its equivalent.

5.1.2.2. Parts shall be immersed for one to two minutes in a solution of 20% HNO_3 - 20% HF - 60% H_2O by volume maintained at room temperature to 125°F. To remove acid, the parts shall then be rinsed in distilled water and acetone alternately.

5.1.2.3. Parts shall be stored as indicated in paragraph 5.1.1.3.

5.1.3. Type 316 Stainless Steel or L-605 Alloys

5.1.3.1. Machined Surface. Parts shall be degreased with acetone or its equivalent.

5.1.3.2. Light Scale. Parts shall be immersed for 10 minutes, or until the surface appears clean and white, in an acid solution of 6 to 15% by volume of HNO_3 (70%) and 0.5 to 1.5% by volume HF (60%) maintained between room temperature and 130°F. To remove residual acid, parts shall be rinsed in distilled water.

5.1.3.3. Parts shall be stored as indicated in paragraph 5.1.1.3.

5.2. Brazing Alloy Placement

5.2.1. Joint components shall be assembled per the engineering drawing.

5.2.2. Parts shall be handled using clean nylon or vinyl gloves.

5.2.3. Brazing alloy powder and the Microbraz binder shall be mixed into a uniform slurry. To form a smooth, even fillet, the braze slurry shall be applied with a spatula. Acetone shall be added to the slurry, as needed, to maintain consistency. After each layer of slurry is applied to the joint, acetone shall be brushed lightly onto the braze fillet until no additional compaction occurs.

5.2.4. The braze slurry shall be applied to the joint until a fillet, approximately twice the size desired after brazing, is produced.

5.2.5. A stop-off compound, Microbraz green or its equivalent, shall be applied by brushing, thus forming circumferential bands on each side of the braze alloy. Utmost care shall be taken to avoid contamination of the braze fillet. If contamination does occur, that portion of the braze

alloy shall be removed with acetone and the procedures outlined in paragraphs 5.2.3. and 5.2.4 shall be repeated.

5.2.6. The joint shall be dried at room temperature for a minimum of four hours or by heating at a maximum of 200°F for two hours.

5.3. Brazing

5.3.1. Temperature Measurements

5.3.1.1. Two Pt/Pt-13% Rh thermocouples shall be attached, by spot tacking, to the L-605 alloy or Type 316 stainless steel components of the joint. These thermocouples shall be placed a maximum of 0.5-inch from the joint.

5.3.1.2. An alternate method may be used whereby one Pt/Pt-13% Rh thermocouple, attached per paragraph 5.3.1.1., and an optical pyrometer shall be used.

5.3.1.3. Pyrometers and temperature measuring equipment shall have been calibrated within the month preceding use.

5.3.1.4. Temperatures recorded per paragraphs 5.3.1.1 or 5.3.1.2 shall not differ by more than 20°F during the brazing temperature cycle.

5.3.2. Brazing Cycle

5.3.2.1. Prepared joints shall be placed in the furnace so that a minimum of 2 inches on either side of the joint interface is within the uniform hot zone of the furnace.

5.3.2.2. Before applying heat to the joint, a vacuum of 1×10^{-4} torr shall be attained. During heat-up, outgassing of binder materials will occur below 1000°F. Short-time (less than one minute) bursts to 1×10^{-3} torr vacuum are allowable. At least 1×10^{-4} torr vacuum shall be maintained when the part is above 1000°F.

5.3.2.3. Furnace temperature shall be increased gradually so that 15 to 25 minutes are required to reach the brazing temperature.

5.3.2.4. The brazing temperature shall be 2160°F, plus or minus 10°F, for 5 to 7 minutes as determined by the higher temperature reading given in paragraph 5.3.1.4.

5.3.2.5. A complete record of time, temperature, and vacuum, signed by the operator, shall be maintained.

6. QUALITY ASSURANCE PROVISIONS

6.1. Inspection. All brazed joints shall be examined visually and by binocular microscope and borescope for the following braze characteristics:

- (1) Cracks,
- (2) Porosity,
- (3) Brazing alloy wettability and flow,
- (4) Uniformity and size of brazing alloy fillets.

These brazing characteristics shall be consistent with good brazing practice.

6.2. Joint Integrity. The brazed joints shall be inspected radiographically; their leak tightness shall be determined by helium mass-spectrometry techniques.

6.2.1. X-ray Inspection

6.2.1.1. X-ray inspection shall be performed in accordance with specification AMS 2635.

6.2.1.2. The radiographs shall be taken in positions that will best delineate lack of brazing alloy flow and porosity.

6.2.1.3. The General Electric project engineer or his representative shall interpret the radiographs and determine braze quality.

6.2.2. Mass Spectrometer Leak Tests

6.2.2.1. The brazed joint shall be evacuated to at least 1×10^{-5} torr by the mass spectrometer vacuum system and subjected to a helium gas stream moved along the joint area.

6.2.2.2. Any leak indication at the maximum sensitivity of the mass spectrometer shall be cause for rejection.

6.3. Repairs

6.3.1. If no cracks or other imperfections have occurred in the base metals, joints may be repaired by repeating the procedures outlined in paragraphs 5.2 and 5.3. Only one additional brazing cycle shall be allowed.

6.4. Columbium Alloy Control Specimens

6.4.1. A columbium alloy specimen, weighing 5 grams or more and taken from the same tubing used to prepare the joint, shall be subjected to all of the thermal treatments applied to the joint. This specimen and a similar, but unheated, specimen of the initial material shall be retained by the Quality Control and Reliability Engineer for subsequent analysis in the event that a determination of the extent of contamination by the vacuum environment is desired.

6.4.2. Chemical analyses of the columbium alloy control specimens shall be conducted for carbon, oxygen, nitrogen, and hydrogen upon the request of the Quality Control and Reliability Engineer. The carbon shall be determined by the induction heating—conductometric method and the other elements shall be determined by the vacuum fusion method. A total increase in the concentration of the above elements, averaged across the tube wall thickness, greater than 100 ppm by weight shall be cause for rejection of the joint. Similarly, an increase in hydrogen content greater than 10 ppm shall be cause for rejection. The following limits shall apply to the analytical results:

Carbon	+	10 ppm
Oxygen	+	50 ppm
Nitrogen	+	50 ppm
Hydrogen	+	2 ppm

SPECIFICATION

OPERATOR CERTIFICATION FOR HIGH TEMPERATURE,
VACUUM FURNACE BRAZING OF BIMETALLIC TUBE JOINTS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

OPERATOR CERTIFICATION FOR HIGH TEMPERATURE,
VACUUM FURNACE BRAZING OF BIMETALLIC TUBE JOINTS

1. SCOPE

1.1. This specification outlines the requirements and procedures for certifying operators to perform high temperature, vacuum brazing of bimetallic tube joints. One component of the joints is a refractory metal or its alloy.

1.2. This specification is applicable to all operator certification under cognizance of SPPS Quality Control.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

SPPS-9
(3 June 1963)

Vacuum Brazing Bimetallic Tube
Joints

FPD B50T56-S1
(23 April 1958)

Cobalt Base Alloy Powder

3. MATERIALS

3.1. Base Metals. The pertinent base metals include columbium, tantalum, or molybdenum or their alloys and 300 series stainless steel or L-605 alloy.

3.2. Brazing Alloy. The brazing alloy shall conform to FPD specification B50T56-S1.

4. EQUIPMENT

4.1. The vacuum furnace shall be a high temperature, cold-wall, vacuum furnace.

4.2. The auxiliary equipment shall be capable of monitoring furnace vacuum and part temperature (plus or minus 5°F).

5. REQUIREMENTS

5.1. Purpose. The qualification tests described here shall determine the operator's ability to produce sound brazements in components and facilities for liquid metal systems. It is intended that the operator demonstrate his capability in vacuum system operation and temperature control of the furnace that will be used in joint fabrication.

5.2. Joint Types. Simple overlap tube joints shall be used.

5.3. Brazing Test Specimens

5.3.1. Base Metals. Any combination of refractory and non-refractory metals given in paragraph 3, as designated by SPPS Quality Control, shall be used.

5.3.2. Tube Joints. The specimen shall have the joint design shown in Figure 1.

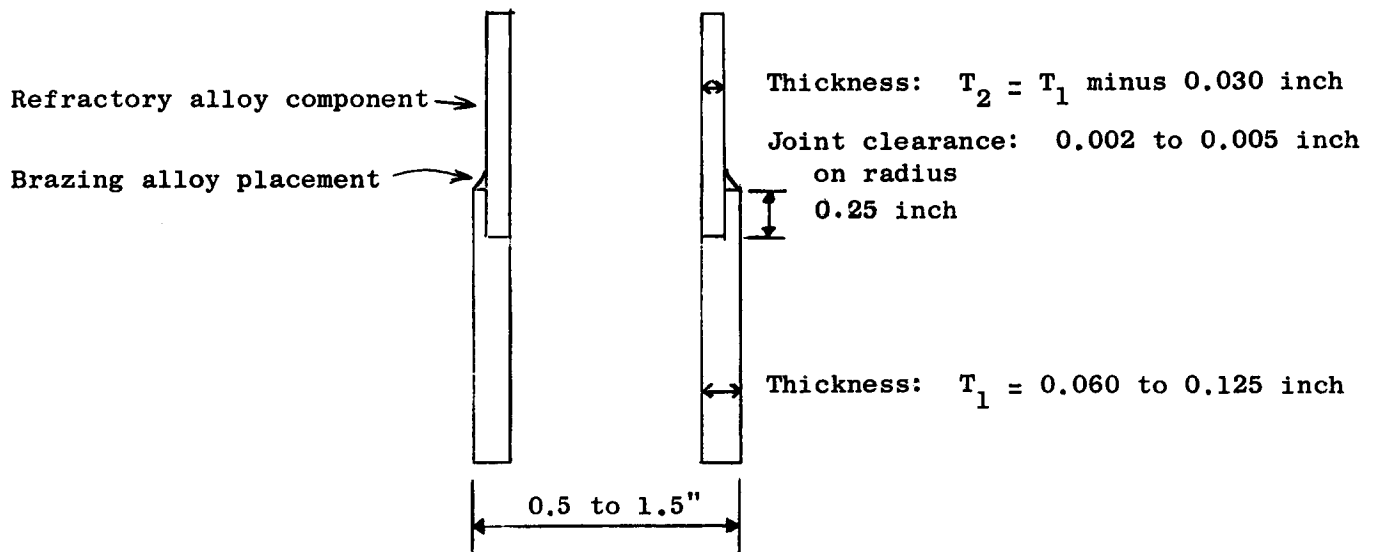


Figure 1. Tube Joint Design.

6. PROCEDURE

6.1. The brazing technique shall conform to specification SPPS-9, paragraph 5.

7. QUALITY ASSURANCE PROVISIONS

7.1. Non-Destructive Testing. The finished brazements shall be inspected by binocular microscope and boroscope for the following characteristics:

- (1) Cracks,
- (2) Porosity,
- (3) Brazing alloy wettability and flow,
- (4) Uniformity and size of brazing alloy fillets.

These brazing characteristics shall be consistent with good brazing practice.

7.2. Destructive Inspection

7.2.1. Two longitudinal sections and one transverse section shall be cut from the brazement for metallographic specimens.

7.2.2. Metallographic specimens shall be mounted and polished using standard metallographic practices. Specimens shall be examined at 50 to 100 X magnification for cracks, porosity, fillet formation, etc. in the as-polished and etched conditions. Microphotographs at 100 X magnification shall be taken on each specimen.

7.3. Records. Copies of the record for each qualified vacuum furnace operator, to be retained by the manufacturer, contractor, or supervisor, shall be available for inspection by the authorized representative of the purchaser or project manager.

SPECIFICATION

CHEMICAL CLEANING OF COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

CHEMICAL CLEANING OF COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

1. SCOPE

1.1. This specification establishes the procedures for the chemical cleaning of columbium and columbium alloy products.

1.2. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements included on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS. None

3. MATERIALS. Materials required under this specification are hydrofluoric acid (48% solution) and nitric acid (70% solution).

4. EQUIPMENT. Necessary equipment includes:

- (1) Acid tank resistant to hydrofluoric/nitric acid mixtures
- (2) Wash tanks
- (3) Cleaning racks resistant to hydrofluoric/nitric acid mixtures
- (4) Safety equipment and clothing.

5. REQUIREMENTS

5.1. The solution required for the proper chemical cleaning of columbium materials is EXCEEDINGLY HAZARDOUS. Extreme caution, which shall be exercised in the preparation and use of the solution, requires that:

- (1) Protective clothing, as specified by the plant safety specialist, shall be mandatory.
- (2) Proper warning signs shall be posted.
- (3) Access to the facility shall be to authorized personnel only.

- (4) An emergency center shall be established and shall store the proper acid antidotes and the equipment and directions for their usage, as specified and demonstrated by the plant industrial hygienist.

5.2. All surfaces of the columbium or columbium alloy products shall be free of grit, metallic chips, grease, oil, fingerprints or any organic markings.

5.3. The cleaned and dried products shall be transferred immediately to polyethylene bags. All hollow parts shall be sealed with suitable non-adherent plugs. Cleaned products shall be handled using clean nylon gloves.

6. PROCEDURE

6.1. The composition of the solution shall be:

Hydrofluoric acid (48% solution)	- 20% by volume
Nitric acid (70% solution)	- 20% by volume
Water	- 60% by volume

The quantity of cleaning solution shall be calculated so that ENTIRE container units of the acids are consumed in the preparation. Acids shall always be added to the water, NEVER the reverse. The prepared solution shall be thoroughly agitated to insure complete mixing and maintained at 100-125°F during pickling.

6.2. The cleaning rack shall be loaded so that the cleaning solution shall have free access to all surfaces of the charge. To assure uniform cleaning, rack loads shall consist of products with as similar dimensions as possible.

6.3. To maintain a minimum storage time for cleaned stock, the chemical cleaning operation shall be scheduled just before any fabrication process. The racked columbium products shall be totally immersed in the solution and agitated by gently raising and lowering the rack beneath the surface of the solution. If hollows (tubes, pipes, cylinders, etc.) are being cleaned, the agitation shall be achieved by moving the rack back and forth in the solution to insure a continuous supply of fresh solution to the internal surfaces. Depending on the mass and configuration of the rack loads, the immersion time will vary. The amount of stock removal required shall be specified by the requestor.

6.4. After the immersion, the rack shall be raised above the solution, tilted slightly and allowed to drain for 2-3 minutes. Then the rack shall be immersed immediately in clean, flowing water and agitated by a back and forth movement of the rack. Immersion in the water rinse shall be no less than thirty minutes with the water at a temperature above

125⁰F. A second rinse shall be conducted for a minimum of five minutes in distilled or de-ionized water at a temperature above 125⁰F. The rack load shall be allowed to drip-dry or shall be dried by blowing dry, clean, oil-free air over and through the load.

7. RECORDS. The part name, date of cleaning, and general observations shall be recorded as specified by the project manager or his representative.

SPECIFICATION

GRIT BLASTING COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

GRIT BLASTING COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

1. SCOPE

1.1. This specification establishes the procedures for grit blasting columbium and columbium alloy products to obtain increased and uniform emittance.

1.2. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements included on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS. None

3. REQUIREMENTS

3.1. Materials. Aluminum oxide powder, classified 280 mesh size and 99% minimum purity, is required.

3.2. Equipment. Necessary equipment includes:

(1) Micro-grit blasting machine with a variable abrasive feed rate and nozzles between 0.018 and 0.125-inch diameter.

(2) Compressed air supply, variable to 80 psig.

3.3. Procedure

3.3.1. The parts shall be degreased, if necessary.

3.3.2. The abrasives shall not be used more than once.

3.3.3. The nozzle shall be kept in motion at all times when the abrasive is in contact with the surface of the parts.

3.3.4. The distance between the nozzle and the surface of the parts shall not be less than 2 inches.

3.3.5. The parts shall be grit blasted using an air pressure of 80 psig; uniform surface finish shall be produced.

3.3.6. Abrasive particles shall be removed from the surface of the parts by wiping with clean lint-free cloths.

4. QUALITY ASSURANCE PROVISIONS

4.1. After grit blasting, parts shall exhibit a uniform surface finish under visual inspection.

4.2. Records. A certificate of conformance shall be supplied with the finished parts. Records shall be maintained by Quality Assurance Personnel.

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE ELECTRON BEAM PROCESS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM
ALLOY BY THE ELECTRON BEAM PROCESS

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for electron beam welding of columbium-1% zirconium alloy. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

AMS 2635
(15 August 1955)

Radiographic Inspection

SPPS-11A
(26 July 1963)

Chemical Cleaning of Columbium
and Columbium Alloy Products

3. REQUIREMENTS

3.1. Equipment

3.1.1. A high voltage electron beam welder, capable of 150 kilovolts at 20 milliamperes, shall be used.

3.1.2. The welding chamber shall be capable of evacuation to less than 5×10^{-5} torr and shall be leak-tight as determined by mass-spectrometer leak checks at a sensitivity of 5×10^{-10} std cc/sec.

3.2. Cleaning

3.2.1. The parts to be joined shall be cleaned in accordance with specification SPPS-11A of 26 July 1963, for material thickness of 0.015 inch or more. Material thicknesses less than 0.015 inch shall be cleaned with reagent grade acetone.

3.3. Fixtures

3.3.1. The fixtures shall hold the parts in proper alignment and shall maintain the gap tolerance listed in Table I.

TABLE I
ALLOWABLE GAP TOLERANCES

<u>Material Thickness</u> <u>(Inch)</u>	<u>Maximum Allowable Gap</u> <u>(Inch)</u>
0.000 to and including 0.010	metal-to-metal contact
0.010 to and including 0.060	0.002
0.60 to and including 0.125	0.003
0.125 and greater	0.005

3.3.2. The fixtures shall be made from materials that do not corrode or rust in air. The fixture components that contact the columbium parts near the weld joint shall be made of molybdenum, tungsten or columbium.

3.3.3. The fixture shall be clean and free of surface contamination.

3.4. Welding Procedures

3.4.1. Welding of components shall be done using equipment and procedures which conform to Section 4. After loading of components and fixtures, the chamber shall be evacuated to 1×10^{-4} torr or better before the filament current is started and to 5×10^{-5} torr or better before the electron beam is activated.

3.4.2. Prior to welding the beam shall be sharply focused upon a tungsten target whose surface is at the same level as the surface of the material to be welded. This procedure may be modified in special cases where surface focusing would not allow complete fusion of the weld joint.

3.4.3. Prior to the first piece welded and subsequent to the last piece welded in each sequence of chamber evacuations, weld bend specimens shall be prepared. Each specimen shall be from 0.04 to 0.08 inch thick, 1 inch wide (minimum), and 2 inches long (minimum) with a longitudinal weld bead weighing 2.5 grams (minimum).

The above specimens shall be tested in accordance with paragraph 4.2. Specimens and samples shall be tagged with the drawing number, part number, and serial number of each part represented by the samples and specimens. These specimens shall be retained by quality assurance.

3.4.4. The completed weldment shall be left in the chamber until sufficient time has elapsed to allow complete cooling of the component to room temperature.

3.4.5. In repair welding, procedures shall be provided by the General Electric project engineer. The component shall then be welded, according to the requirements of this specification, and re-inspected.

3.5. Postweld Heat Treatment

3.5.1. The weld and adjacent heat-affected zone shall be postweld₋₅ annealed at 2200°F, plus or minus 25°F, for one hour in a vacuum of 1×10^{-5} torr or better. The annealing may be local at the weld joint or the entire weldment may be annealed in a vacuum furnace. The procedures and equipment used shall conform to paragraph 4.3.

3.5.2. Localized heating shall be done using a heater having only refractory metals in the hot zone. Dense vitrified alumina (99.2% purity) shall be used as the heater electrical insulation in the cold zone of furnace. The temperature of the weld joint shall be measured by a Pt/Pt-Rh thermocouple that is resistance spot-tacked adjacent to the weld metal. After heat treatment the thermocouple spot-tack shall be filed off.

3.5.3. A columbium-1% zirconium control specimen of 0.040 to 0.080-inch thick sheet, weighing 5 grams or more, shall accompany the welded components or parts through postweld vacuum heat treatment.

The above specimens and a similar, but unheated, specimen of the control material shall be submitted with the finished parts. Specimens shall be tagged with the drawing number, part number, and serial number of each part represented by the specimens.

Note: These specimens shall be retained by quality assurance for subsequent chemical analysis, should an investigation of vacuum environment contamination be desired.

3.6. Defect Limits. The completed weldment shall be examined for defects visually and by ultrasonic or X-ray techniques, or both. Welds which do not meet the following quality requirements of this specification are unacceptable.

3.6.1. Visual and Radiographic. Welds shall be reasonably smooth and uniform in appearance and free of the following defects by visual and radiographic inspection:

1. Cracks in the weld and adjacent base metal
2. Crater checks or cracks
3. Surface inclusions and pores
4. Undercutting of the base metal
5. Depression of the weld below the base metal
6. Unfilled weld craters or stops
7. Damage to the weld and adjacent base metal by contamination from foreign materials or metals
8. Lack of fusion of the weld deposit to the base metal
9. Incomplete fusion and penetration to the root of the joint (back side of butt and groove welds)
10. Porosity in the weld metal

3.6.2. Ultrasonic. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication. For tubing, inside diameter (or under side) defects shall be compared to the indication from the notch on the inside diameter (or under side), and outside diameter (or upper side) defects shall be compared to the indication from the notch on the outside diameter (or upper side). Indications less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the upper side (or outside diameter) calibration indication. Defects more than half the distance from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter (or under surface) notch.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by X-ray or ultrasonic techniques, or both.

4.1.1. X-ray Inspection

4.1.1.1. X-ray inspection shall be done according to AMS 2635, "Radiographic Inspection". Fine grain film shall be used for maximum sensitivity. The penetrameter thickness and hole sizes shall be based on the smaller section thickness being joined. The radiographic technique shall be considered satisfactory if the smallest hole in the penetrameter can be clearly distinguished on the radiograph.

4.1.1.2. The radiographs shall be taken in positions that will best delineate lack of weld penetration and fusion. Wherever possible, the film shall be placed on the back side of the weld joint.

4.1.1.3. When the back side of the weld is not accessible and the radiation must pass through a double thickness, only that portion of the weld next to the film shall be considered.

4.1.1.4. Radiographic interpretation for weld quality shall be done by the General Electric project engineer or his representative.

4.1.2. Ultrasonic Inspection

4.1.2.1. When required by the drawing, welded joints shall be ultrasonically rather than radiographically inspected.

4.1.2.2. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Shear wave technique shall be used with the beam traverse perpendicular to the weld centerline. Inspection shall be from both directions toward the weld. For welds in tubing, a transducer focused to the diameter of the tubing shall be used with a maximum axial length on the transducer of 0.5 inch. For welds in plate, a spherically focused transducer or a cylindrically focused transducer of less than 1.5 inch diameter focus and 0.5 inch or less long shall be used with its axis parallel to the weld centerline.

4.1.2.3. Calibration shall be on notches cut in a tube or plate of similar material, preferably near a weld of similar nature. These circumferential notches shall be: depth, 3% of the original wall or plate thickness; width, not more than the depth; length, at least one beam width. One notch shall be placed on the inside diameter or under side near the weld; the other notch shall be placed on the outside diameter or upper side near the weld but on the opposite side of the weld or at least two beam widths away (at the same distance from the weld) if on the same side of the weld. Focusing shall be done to maximize the indication from the notch on the under side or inside diameter. After focusing is completed, whichever indication is larger shall be set at 80% and the amplitude of the indication from the other notch shall be recorded. Distance corresponding to the wall or plate thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. All testing since previous calibration shall be repeated whenever the calibration gain changes by more than 10%.

4.2. Welding Equipment and Procedure

4.2.1. Before welding actual columbium alloy parts and components, the welding equipment and procedures shall be qualified by welding columbium-1% zirconium alloy and testing as follows.

4.2.2. A full penetration bead on sheet weld shall be made in the electron beam facility in the columbium-1% zirconium alloy. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch.

4.2.3. Four strips shall be cut from the welded joint with the weld running transverse to the length. The minimum width of three strips shall be four times the base metal thickness or 1/2 inch, whichever is larger,

4.2.4. Three welded strips shall be bend tested using a 75-degree V-block and mating V-punch with a radius three times the base metal thickness. Each weld is to be placed face down and centered under the punch radius. The reinforcement on the back side shall be removed before testing.

4.2.5. All three test strips shall withstand a transverse bend through 90 degrees without cracking. The weld and heat-affected zones shall be visually examined for cracks at a magnification of 10X. All three specimens must pass the bend test requirements.

4.2.6. Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the fourth strip. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. These chemical analyses shall demonstrate that the degree of environmental contamination did not exceed the following limits: increase in oxygen content--less than 50 ppm; increase in nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results.

Carbon	±	10 ppm
Oxygen	±	30 ppm
Nitrogen	±	30 ppm
Hydrogen	±	2 ppm

4.3. Postweld Annealing Procedures and Equipment

4.3.1. Before annealing welded joints in components, the annealing procedures and equipment shall be qualified as described to insure that the weldments are not contaminated and embrittled during the operation.

4.3.2. A sample welded joint simulating the actual weldment shall be welded using qualified equipment and procedures. The weld shall be heat-treated at 2200 F, plus or minus 25 F, for one hour in a vacuum using the procedures and equipment that will be used for production welds. If localized heat-treating equipment is used, the temperature of the weld shall be measured by a Pt/Pt-Rh thermocouple which is resistance spot-tacked to the part.

4.3.3. Three strips shall be cut from the joint. Two shall have a width four times the base metal thickness but not less than 1/2 inch. The third one shall be sufficiently wide to obtain a weld metal, chemical analysis sample. The weld shall run transverse to the length of the strip at midpoint.

4.3.4. One transverse weld bend strip shall be bend tested as annealed, and the other shall be bend tested after aging at 1500°F for fifty hours in a vacuum furnace at a pressure of 1×10^{-5} torr maximum. The bend test procedures shall be those described in paragraph 4.2. Both bend strips must pass the test requirements.

4.3.5. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the third strip. These analyses shall demonstrate that the degree of environmental contamination did not exceed the limits set forth in paragraph 4.2.

4.3.6. Qualifying both welding and annealing procedures and equipment simultaneously is permissible. The test results must meet the requirements of paragraphs 4.2. and 4.3.

4.4. Reports. A report shall be submitted with the finished parts and shall include all vacuum, temperature, and inert gas purity measurements applicable to welding and heating, the X-rays of the weld joints, and/or when required, location and approximate depth of ultrasonic indications found. Ultrasonic reports shall also include the ultrasonic equipment and transducer used and calibration data. The above reports and all accompanying X-rays, charts, etc. shall reference the drawing number, part number, and serial number of each part represented and, when multiple welds on a part are made in separate environments, the particular weld to which the information is applicable.

Note: These reports shall be retained by quality assurance, should subsequent weld evaluation be desired.

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SPECIFICATION

MASS SPECTROMETER LEAK DETECTION USING HELIUM

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

Mass Spectrometric Leak Detection

- CONTINUED

DATE

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03-0013-00-B

1. SCOPE

1.1. Scope. This specification describes the helium mass spectrometric method and quality requirements for leak detection in various types of containment equipment. Reference to this specification shall be made on engineering drawings whenever applicable, however, information or requirements on such drawings supercede this specification.

1.2. For other methods of leak detection, see MIL-STD 271C (Ships).

2. APPLICABLE DOCUMENTS2.1. Government Documents

MIL-STD 271C (Ships)
1 October 1963

Non-Destructive Testing

2.2. Non-Government Documents. None

3. REQUIREMENTS3.1. Equipment and Materials

3.1.1. Leak Detector. Final acceptance testing shall be accomplished using a mass spectrometric helium leak detector. Appropriate conversion factors are presented in Table I.

Table I

Leakage Units (Flow)

<u>To Convert From</u>	<u>To</u>	<u>Multiply By</u>
Std. cc/sec.	Micron-liters/sec.	7.6×10^2
	Micron cubic feet/hr.	9.66×10^4
	Torr-liters/sec.	7.60×10^{-1}
Micron-liters/sec.	Std. cc/sec.	1.32×10^{-3}
	Micron cubic feet/hr.	1.27×10^2
	Torr-liters/sec.	1.00×10^{-3}
Micron cubic feet/hr.	Std. cc/sec.	1.04×10^{-5}
	Micron-liters/sec.	7.87×10^{-3}
	Torr-liters/sec.	7.87×10^{-6}
Torr-liters/sec.	Std. cc/sec.	1.32
	Micron-liters/sec.	1.00×10^3
	Micron cubic feet/hr.	1.27×10^5

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3.1.2. Sensitivity of the Instrument

3.1.2.1. This test, which serves to calibrate the instrument only, shall be performed with a calibrated leak attached directly to the leak detector with helium applied at atmospheric pressure. Recalibration will be performed upon change in setup or after moving the leak detector from one place to another. The sensitivity of the instrument shall be determined as follows:

3.1.2.2. Prior to leak detection (during calibration) random meter movement (oscillations) shall be less than 10% of the full scale value at the maximum sensitivity (the times 1 scale or equivalent) of the instrument.

3.1.2.3. During the calibration, the length of time for the output meter to go from zero to full scale on any sensitivity range shall not exceed one second.

3.1.2.4. The operator will assure himself that the output meter has risen to a maximum, constant level to be sure that equilibrium has been reached in the system.

3.1.3. Calculation of Sensitivity. The sensitivity shall be calculated as follows:

$$S = \frac{L}{CHD}$$

where

S = Sensitivity

L = Calibrated leak (micron cubic feet per hour or standard cubic centimeters per second of air)

CHD = Deflection caused by helium during calibration
(calibration helium deflection)

3.1.4. Helium. The helium used shall be a pure commercial grade which is not oil pumped.

3.2. Preliminary Conditions

3.2.1. Stages for Helium Leak Testing. When any part of a test piece cannot be reached for proper repair after final assembly, leak testing shall be performed during appropriate predetermined stages of fabrication.

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3.2.2.1. One or both of the following tests may be conducted on the test piece during or after assembly to locate large leaks in order to reduce time for leak testing with the helium mass spectrometer.

3.2.2.2. The test piece shall be subjected to an internal gas pressure (air or inert gas, not helium) not to exceed the design pressure. All connections and welded joints shall then be examined for leaks by application of a soap solution or its equivalent. The joint or connection will be observed for the growth in size of bubbles or the obvious removal of the soap solution by the force of the gas through the leak.

3.2.2.3. Vacuum leak testing shall be performed with the test piece, including all welds and joints, unpainted and free of any type of coating or filler. The test piece shall be evacuated with the leak detector vacuum system to less than 300 microns of mercury. Where a pressure greater than 300 microns exists, an attempt will be made to reduce the pressure by tightening all connections and coating welds with a compound such as Apiezon "Q" putty. The location of large leaks will be noted when a large decrease in pressure occurs. Careful examination of the suspected area will usually disclose the defect, and repair procedures can be implemented. When a pressure of 300 microns or less can be attained, helium leak testing can be initiated.

3.2.3. Cleanliness. Whenever possible, the test piece shall be clean and free of water vapor, oil, grease and other contaminants which interfere with the mass spectrometer leak test.

3.2.4. Adsorbed Gases. Inert gases or inert gas mixtures used during fabrication should not contain helium. When fabrication procedures make it necessary to use helium the test piece shall be evacuated below a pressure of 50 microns. Whenever possible, test pieces will be warmed (150-250° F or more) to remove adsorbed gases to reduce the background within the leak detector to an insignificant level. On a relatively leak-free system, an insignificant background is indicated by an output reading on the most sensitive scale which is steady to the extent that the needle oscillations are less than 10% of the full scale deflection. In less ideal cases, where the output is high and/or unsteady due to previous exposure to helium, high outgassing rates or relatively large leaks, leaks may be detected; but the significance of a particular reading must be based on the interpretation of the operator.

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3.2.5. Openings in the Test Piece. All unused openings in the test piece shall be sealed leak-tight. All leaks around these closures shall be sealed, using sealing wax, cements or other suitable material which can be readily and completely removed after testing.

3.2.6. Vacuum. The test piece shall be evacuated to 50 microns or less as determined by a thermocouple gauge, or its equivalent, which shall be placed in the vacuum line immediately adjacent to the test piece. If, after all preliminary testing, with all openings leak-tight, a system can be evacuated to only 300 microns, the leak detector may then be used with the following technique. The operator can determine if the instrument has enough sensitivity during leak checking to detect the leak. When the leak is detected and repaired, the test piece shall then be evacuated to 50 microns or less and rechecked, using the helium leak detector as described in paragraph 3.5.

3.3. Frequency of Calibration. The leak detector must be calibrated at the beginning and completion of any leak test, at least, and more frequently if the leak testing is prolonged.

3.4. Calculation of Actual Leak Rate. Leak rates shall be calculated as follows:

$$LR = S \times THD$$

where LR = Leak rate (std. cc/sec. or micron cubic feet/
hour air)

S = Sensitivity

THD = Deflection caused by helium during leak checking
(test helium deflection)

This calculation applies only when the leak is exposed to helium at one atmosphere pressure and after the indicated level indication has risen to a maximum, constant value. In this specification it applies only to the Envelope Method described below.

3.5. Helium Leak Testing Methods

3.5.1. Envelope Method. The envelope method of helium leak detection shall be used to indicate the total leakage of the item (including portions of test pieces) after all large leaks have been repaired as indicated by an ability to evacuate the test piece to a pressure lower than 50 microns. The procedure is as follows:

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- a. Interconnect the calibrated leak detector with the item under test.
- b. Evacuate to 50 microns or less as measured by a thermocouple gauge or its equivalent placed in the evacuation line adjacent to the test piece
- c. Envelop the entire test piece or portion thereof with a suitable container such as a plastic bag.
- d. Fill the space between the outside of the item under test and the container with helium at, or slightly above, atmospheric pressure.
- e. With the inlet valve to the detector in a wide open position, the pumping speed of the detector reduced to a minimum, and no auxiliary pumping on the test piece, the leakage into the detector is measured on the output meter. This measurement of leakage is valid only after the deflection has risen to a steady, constant value.

3.5.2. Probe Method. When the actual leak rate exceeds the permissible value or when an over-all probe test is specified, all welds or other suspected areas shall be inspected by means of a fine jet of helium on one side of the weld or other joint and the leak detector on the other. After repair of all defects located by this method, the actual leak rate will be evaluated, if required, using the Envelope Method (paragraph 3.5.1.).

3.5.3. Sniffer Method. Where configuration or access makes probe or envelope testing impossible, the system under test will be pressurized with helium to a point within design specifications. All welds or suspected areas shall be inspected by the use of an evacuated probe which will draw helium from a leak into the leak detector. All leaks detected by this method must be repaired since it is not possible to measure the size of the leak using this method.

3.6. Allowable Leakage Rates

3.6.1. Categories. Two categories of acceptable leakage rates are specified for various applications: critical and non-critical.

3.6.2. Critical Applications. In critical applications a maximum total leakage rate of 5.0×10^{-10} std. cc/sec. of air is specified unless otherwise noted on engineering drawings or directed by the design engineer.

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3.6.3. Non-Critical Applications. When non-critical applications are noted on engineering drawings, the maximum, total, allowable leak rate will be 5.0×10^{-8} std. cc/sec. of air.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of a helium leak test on a piece of test equipment or component, the leak detector operator will prepare a Helium Leak Test Process Control Record (Form SP1067) in duplicate. He will send the original to the person requesting the leak test and will retain the duplicate for the Helium Leak Test Record notebook.

5. PREPARATION FOR DELIVERY. Not applicable

6. NOTES. None

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SPECIFICATION

WELDING OF AUSTENITIC STAINLESS STEELS

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

WELDING OF AUSTENITIC STAINLESS STEELS

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NO.

03-0014-00-B

1. SCOPE

1.1. Scope. This specification covers the requirements for welding austenitic stainless steels in the fabrication of austenitic stainless steel components, facilities, and test devices.

2. APPLICABLE DOCUMENTS2.1. Government Documents

MIL-STD-271C (Ships)
(1 October 1963)

Non-Destructive Testing Requirements
for Metals

2.2. Non-Government Documents

ASME Boiler and Pressure
Vessel Code, 1962 and
Addenda

ASTM A371-62T
(1962)

Tentative Specification for Corrosion-
Resisting Chromium and Chromium-Nickel
Steel Welding Rods and Bare Electrodes
(AWS A5.9-62T)

ASTM A298-62T
(1962)

Tentative Specification for Corrosion-
Resisting Chromium and Chromium-Nickel
Steel Covered Electrodes (AWS A5.4-62T)

ASTM B297-55T
(1955)

Tentative Specifications for Tungsten
Arc-Welding Electrodes (AWS A5.12-55T)

ASTM E165-63
(1963)

Standard Methods for Liquid Penetrant
Inspection

ASTM E142-64
(1964)

Controlling Quality of Radiographic
Testing

ASTM E94-62T
(1962)

Tentative Recommended Practice for
Radiographic Testing

AWS A2.0-58
(1958)

Standard Welding Symbols (American
Welding Society)

AWS A3.0-61
(1961)

AWS Definitions-Welding and Cutting
(American Welding Society)

3. REQUIREMENTS

3.1. Operator Qualification. When the components, facilities, or piping are to be fabricated to a specific code, standard, or specification, the welders shall be qualified for proficiency as required by the applicable documents. The applicable documents

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shall be specified on the engineering drawings, specifications, or purchase order. When no code, standard, or specification is specified, the components shall be fabricated by welders who are proficient in the welding of austenitic stainless steels by the applicable welding process.

3.2. Welding Processes and Equipment

3.2.1. Welding Processes. The welding process used shall be that designated for the joint on the engineering drawing by welding symbols. The process designations are:

P8G - Gas tungsten arc

P8J - Gas metal arc

P8B - Shielded metal arc

3.2.2. Gas Tungsten Arc. Welding by this process, manual or automatic, shall be done using direct current, straight polarity. The torch gas cup shall be of adequate size to provide optimum gas coverage to the weld zone. Tungsten electrodes shall conform to ASTM B297-55T and shall be Class EWTh-2.

3.2.3. Gas Metal Arc. The welding equipment shall be in good condition and free of water and gas leaks. The gas shielding cup shall be of adequate size to provide optimum gas coverage to the weld zone.

3.2.4. Atmospheres. For gas tungsten arc welding, argon of 99.95% minimum purity shall be used for both torch shielding and weld joint backup protection. For gas metal arc welding, argon-1% oxygen shall be used through the welding head and 99.95% purity argon shall be used to protect the back side of the weld.

3.2.5. Welding Filler Wires and Electrodes

3.2.5.1. The welding filler wire and/or electrode shall be that specified on the engineering drawing, part specification, or welding procedure specifications. Root pass inserts, if used, shall be manufactured from material meeting the specifications for the welding filler metal.

3.2.5.2. Bare welding wires for P8G and P8J welding processes shall conform to ASTM A371-62T. Covered welding electrodes for the P8B process shall conform to ASTM A298-62T.

3.2.5.3. Coated welding electrodes used for shielded metal arc welding shall be purchased in commercial moisture-proof containers. Electrodes that have been exposed to ambient atmosphere for more than eight hours shall be baked at 350°F for two hours prior to use to drive off absorbed water vapor in the coating.

3.2.5.4. When the welding wire or electrode is not specified as described in paragraph 3.2.5.1., it shall be selected from Table I for the alloys to be joined.

3.3. Fabrication Techniques

3.3.1. Gas Shielding

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TABLE IAUSTENITIC STAINLESS STEEL WELDING FILLER MATERIALS

Alloys to be welded	302 304 308	304L	309	310	316	316L	321
302, 304, 308	ER308 E308	ER308L E308L	ER308 E308	ER308 E308	ER316 E316	ER316L E316L	ER316 E316
304L		ER308L E308L	ER308L E308L	ER308L E308L	ER316L E316L	ER316L E316L	ER316L E316L
309			ER309 E309	ER309 E309	ER316 E316	ER316L E316L	ER316 E316
310				ER310 E310	ER316 E316	ER316L E316L	ER316 E316
316					ER316 E316	ER316L E316L	ER316 E316
316L						ER316L E316L	ER316L E316L
321							ER321

Note: "ER" designates bare wire for P8G or P8J welding processes. "E" designates coated electrodes for P8B welding process.

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3.3.1.1. Adequate shielding against air drafts shall be provided for all welding operations to avoid disturbance of the torch gas shielding and/or the welding arc.

3.3.1.2. The back side of the weld joint shall be protected by argon shielding in a manner that will protect the joint against contamination and oxidation.

3.3.1.3. All weld joints in pipe, tubing, tanks, and containers shall have the back side of the weld protected by argon. The interior of the pipe or container shall be purged of air at the joint by sealing off the interior on both sides of the joint or both ends of the pipe or container. No welding shall be done until the interior has been purged by a volume of argon equal to five times its volume. Systems already pressurized with argon need not be purged.

3.3.2. Joint Preparations

3.3.2.1. The edges of the parts shall be grooved for welding by machining, grinding, or filing as shown on the engineering drawing or specifications for the particular joint design and thickness.

3.3.2.2. When the surfaces to be welded are ground, the grinding wheel and techniques used shall not leave imbedded grit particles in the stainless steel.

3.3.2.3. The edges and surfaces to be joined and the adjacent base metal (about 2" on each side of the joint) shall be cleaned of all oil, grease, dirt and other foreign matter. The areas shall also be free of surface oxides and scale. No residual cleaning compounds shall be left on the surfaces.

3.3.2.4. Before applying weld metal on the second side to be welded, the root of double welded joints shall be prepared by machining, grinding, or filing to sound metal, free of cracks, laps, seams, and craters. The root of the weld shall be liquid penetrant inspected for defects. All inspection residue shall be removed before starting to weld.

3.3.3. Joint Positioning

3.3.3.1. Fixturing. The joints to be welded shall be positioned to provide proper alignment, match of parts, and root opening.

3.3.3.2. Alignment. The joint edges shall not be misaligned more than 20% of the thinner section being joined, or 1/16", whichever is less. When abutted ends of unequal internal diameters exceed the allowable misalignment, the part with the smaller inner diameter shall be machined to match that of the larger diameter. The internal taper shall not exceed 1:4.

3.3.3.3. Tack Welds. After the parts are properly positioned, tack welds may be used to maintain alignment during welding. The tack welds shall be cleaned of oxide with a stainless steel wire brush prior to making the first pass. Cracked tack welds shall be removed and retacked. Tack welds shall have complete fusion and penetration to the weld joint root.

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3.3.3.4. Weld Inserts. Consumable weld inserts are permissible provided the welding procedures used insure complete consummation of the insert in the root pass. Backing rings shall not be used unless (1) specifically called out on the drawing or specifications, or (2) they are machined away after welding.

3.3.4. Welding

3.3.4.1. Each joint shall be made using accepted welding procedures for the particular joint type, position, and thickness of base metal.

3.3.4.2. All weld metal that will be in contact with liquid metals contained in test apparatus shall be deposited by P8G or P8J process. In multipass welds, the first two passes shall be deposited by the P8G or P8J process. Shielded metal arc welding (P8B) may be used for fill passes of the joints, except for welds joining Type 321 stainless steel. Welds joining Type 321 stainless steel must be made using either P8G or P8J process for all passes. The welds must meet the quality requirements of section 3.4.

3.3.4.3. Each weld pass shall be thoroughly cleaned with a stainless steel wire brush to remove surface oxidation. Weld deposits shall be free of flux, oxide, dirt, welding defects, etc. before welding over them or connecting to them.

3.3.4.4. Preheating shall not be used.

3.3.4.5. Interpass temperature shall not exceed 500°F.

3.3.4.6. Completed welds shall be cleaned and wire brushed for visual inspection.

3.4. Quality Requirements

3.4.1. General. Weld deposits shall be reasonably smooth and uniform in appearance, have complete fusion, and blend smoothly into the base metal. The welded joints shall be free of the following defects by any and all methods of inspection:

1. Cracks of any type or size in the weld and adjacent base metal,
2. Crater checks and cracks,
3. Surface holes,
4. Cold laps in and along the edge of the weld,
5. Overlap of weld metal on the base metal,
6. Undercutting along the edges of the weld or depression of the weld face below the adjacent base metal,
7. Weld craters,
8. Damage to the weld metal by oxidation,
9. Incorrect weld profile and size,
10. Lack of complete (100%) joint penetration in groove welds,
11. Incomplete fusion between weld metal and/or base metal.

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3.4.2. Drop Through. Weld metal on the back side of the joint shall not protrude above the base metal surfaces by more than 25% of the base metal thickness or 1/16", whichever is the least.

3.4.3. Groove Weld Reinforcement. Weld reinforcement on the face side of the joint shall not exceed the following:

Base Metal Thickness (inch)	Height of Reinforcement (inch)
Up to 1/2	1/16
Over 1/2 to 1	3/32
Over 1 to 2	1/8

3.4.4. Fillet Weld Contour. The face of a fillet weld shall be at approximately equal angles to the sections it joins, unless specifically noted otherwise on the drawing. The weld face may be slightly convex, flat, or slightly concave. Convex welds shall have a maximum convexity of 0.1 S plus 0.03 inch where S is the average length (inch) of the two legs of the fillet weld.

3.4.5. Radiographic Welds - Porosity and Inclusions

3.4.5.1. The total area of porosity and inclusions, as determined from the radiographic film, shall not exceed 0.03T square inch in any six-inch length of weld, where T is the average thickness of the weld. If the weld joint is less than 6 inches long, the total allowable area of porosity and inclusions is reduced in proportion of weld length, inches/6.

3.4.5.2. The largest dimension of any single indication shall not exceed 0.15T or 3/32 inch, whichever is smaller.

3.4.5.3. In any 2T length of weld, indications of porosity and inclusions may be clustered to a concentration four times ($0.04T^2$) that permitted in paragraph 3.4.5.1. Such indications shall be included in the total area permitted in paragraph 3.4.5.1. including all of the cluster.

3.4.5.4. Any group of aligned indications having a summation of diameters greater than T in a weld length of 24T is unacceptable. If the weld joint is less than 24T long, the allowable summation is reduced in proportion to weld length/24T.

3.4.5.5. Any two indications separated by a distance less than six times the largest dimension of the larger indication is unacceptable.

3.4.6. Leaks in Welds

3.4.6.1. Each welded joint tested by hydrostatic, gas pressure, or pressure rise (vacuum) methods shall show no evidence of leakage.

3.4.6.2. Each welded joint tested using a helium mass spectrometer, shall have a leak rate of less than 5×10^{-10} standard cc/sec of air.

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4. QUALITY ASSURANCE PROVISIONS

4.1. Visual Inspections. Visual inspections and measurements of the parts to be joined, joint preparation and alignment, and the welded joint shall be performed as required.

4.2. Radiographic Inspection

4.2.1. Applications. Welded joints shall be radiographed for soundness when radiographic inspection (RT) is called out on the engineering drawing or parts specification. Welds made in existing facilities and components that contain liquid metals at 300°F and above will be radiographed provided the radiographs will adequately delineate the welded joint. Radiographic procedures shall conform to ASTM E94-62T, Tentative Recommended Practices for Radiographic Testing.

4.2.2. Radiographic Quality Control. Radiographic quality control procedures used shall be those described in the specification called out on the engineering drawing or parts specification. If no specification is called out, the procedures described in the specifications listed below shall apply to the applicable types of components. Weld quality requirements are given in Section 3.4.

ASTM E142-64: Test components and equipment

ASME Boiler and Pressure Vessel Code: Facilities installations and components including piping, vessels, tanks, boilers, condensers, heat exchangers, and valves.

4.2.3. Penetrameters. Penetrameters shall be used for all radiographs and the penetrameter image will be employed to determine the radiographic quality. Penetrameter design and material shall be that given in the applicable specification.

4.2.3.1. Penetrameter Placement. Penetrameters shall be employed as required by the applicable specification. Normally, the penetrameter is placed on the source side of the weldment. When it is not practical to place the penetrameter on the section being examined, and in the plane normal to the radiation beam, it may be positioned on a block of radiographically similar material placed as close as possible to the area being radiographed. The block shall be the same thickness as the total weld thickness, and it shall be placed so that the penetrameter is at the same distance from the film as if it were placed on the source side of the weld joint being radiographed.

4.2.3.2. Double-Wall Radiography. When radiographing double-wall welds, such as joints in pipe or tubing, the thickness of the penetrameter employed shall be based upon the total thickness of material between the penetrameter and the film.

4.2.4. Weld Surface Preparation. Accessible surfaces of welds shall be prepared as necessary so that valleys between beads, weld ripples, and other surface irregularities are blended so that radiographic contrast due to surface condition cannot mask or be confused with that of any defect.

WELDING OF AUSTENITIC STAINLESS STEELS

- CONTINUED

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4.2.5. Pipe and Tube Joints. Unless otherwise specified on the engineering drawing, parts specification, or purchase order, welds in pipe and tube of 3 inches O.D. and smaller may be radiographed by the double wall technique and the weld zones in each wall viewed for acceptance. A minimum of two radiographs shall be taken 90° to each other. For welds in pipe and tubes having an O.D. larger than 3 inches, only the weld zone closest to the film shall be viewed for acceptance. A minimum of four radiographs shall be taken 90° to each other.

4.2.6. Source to Film Distance. Minimum recommended radiation source to film distance d_o (inches) is that calculated by the following formula:

$$d_o = 2.5 Ft$$

where F = maximum effective radioisotope source or focal spot dimension, mm.
 t = weld thickness, or pipe or tube O.D., inches

Where a gap exists between the weldment and the film holder, the minimum source to film distance should be increased by the ratio of:

$$\frac{t + \text{gap}}{t}$$

4.2.7. Identification of Radiograph Films. A system of positive identification of the film shall be used. The following information shall appear on each radiograph and in the records accompanying each film:

1. Organization making the radiograph.
2. Date of exposure.
3. Identification of (a) the component, facility, or system, (b) the weld joint, and (c) the viewing direction.
4. The radiograph is the original area or a repaired area.

4.2.8. Radiographic Quality Level. Unless otherwise specified on the engineering drawing or parts specification, the radiographic quality level shall be 2% (2+2T) or better.

4.2.9. Interpretation of Weldment Radiographs. The radiographs shall be examined for quality and for unacceptable weld defects described in paragraph 3.4. Final interpretation of radiographed welds for conformance to quality requirements is reserved by the General Electric Company.

4.2.10. Disposition of Radiographs. Radiographs of weldments shall be forwarded to SPPS Quality Control for filing, unless the fabricator is required to retain the radiographs by law or other instructions.

4.3. Liquid Penetrant Inspection

4.3.1. Application. Welds shall be liquid penetrant inspected for soundness when liquid penetrant inspection (PT) is called out on the engineering drawing or parts specification. Welds made in existing facilities and components that contain liquid metals at 300°F and above, and cannot be radiographed in a suitable manner to show weld defects, shall be liquid penetrant inspected.

WELDING OF AUSTENITIC STAINLESS STEELS

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4.3.2. Specifications. The applicable inspection specification shall be that called out on the engineering drawing or parts specification. If none is called out, the specification ASTM E165-63 shall apply for the particular process.

4.3.3. Surface Condition. As-welded surfaces, following the removal of surface oxides, or flux, shall be considered suitable for inspection without grinding. Shot, sand, grit, and vapor blasting shall not be done on surfaces to be inspected.

4.3.4. Penetrants and developers shall be sulfur free types.

4.3.5. Multiple Pass Welds. When more than one pass of weld metal is required to complete a weld joint, each layer of weld metal shall be inspected for defects. All indications shall be considered as described in paragraph 4.3.6. Prior to further welding, all penetrant and developer shall be cleaned from the joint and adjacent areas using a suitable solvent.

4.3.6. Relevant and Irrelevant Indications. All indications in the weld and along the fusion line between the base metal and weld metal are considered relevant and shall be further evaluated or repaired. The indication shall be explored by removing the surface roughness or other conditions believed to have caused the indication to determine if defects are present. The weld shall be reinspected. Any indications are considered relevant and shall be evaluated with respect to quality requirements of paragraph 3.4.

4.4. Leak Testing

4.4.1. Applications. Weldments shall be checked for leakage as specified on the engineering drawing or parts specification. All welds made in existing components and facilities that will contain liquid metal shall be leak checked.

4.4.2. Specifications. When leak testing using a helium mass spectrometer is required and no testing specification is given, the testing shall be done as described in MIL-STD-271C (Ships).

4.4.3. A final leak check shall be performed on the completed weld assembly using the specified method.

4.5. Surveillance. Representatives of the General Electric Company shall be permitted free access for inspection of welding operations, fabrication, materials, and testing. The vendor shall afford each representative all reasonable facilities to satisfy himself that the fabrication is being furnished in accordance with the specified requirements.

4.6. Rejection of Weldments. Weldments and components not conforming to the specifications shall be rejected.

5. PREPARATION FOR DELIVERY. Not applicable.

6. DEFINITIONS

WELDING OF AUSTENITIC STAINLESS STEELS

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6.1. Welding Symbols. Interpretation and meaning of welding symbols on engineering drawings and specifications are those in AWS A2.0-58 Standard Welding Symbols, unless specifically delineated otherwise on the drawings and specifications.

6.2 Welding Terminology. Welding terms in this specification are defined in AWS A3.0-61.

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SPPS-50
30 April 1964
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SPECIFICATION

ARC WELD GROOVE DESIGNS FOR AUSTENITIC STAINLESS STEELS, L-605,
COLUMBIUM, AND TANTALUM ALLOYS

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

SPECIFICATION

ARC WELD GROOVE DESIGNS FOR AUSTENITIC STAINLESS STEELS, L-605, COLUMBIUM, AND TANTALUM ALLOYS

1. SCOPE

1.1. Scope. This specification defines the typical groove designs for inert gas shielded tungsten arc welded joints in liquid metal containment components, assemblies and facilities. It is applicable to austenitic stainless steels, L-605, columbium, and tantalum alloys.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents. Bulletin No. 214 - Specific Safety Requirements Covering the Installation of Pressure Piping Systems, Industrial Commission of Ohio, 1961. ASME Boiler and Pressure Vessel Code, 1962.

3. REQUIREMENTS

3.1. Joint Designs. The weld groove designs are shown in Figure 1. The groove dimensions for the four materials are tabulated. The groove designs are typical and they are applicable to most weldment designs.

3.2. Applications. The groove designs are to be utilized in the design of liquid metal containment facilities, components, and test devices. When welding procedures development shows the need for special groove designs to obtain the required weld quality, the modified design shall be incorporated on the engineering drawing with approval of SPPS Materials and Processes engineering and design engineering.

3.3. Groove Preparation. The edge preparation shall be machined, using procedures that will not (1) induce flaws in the metal, (2) leave embedded particles or other foreign matter on the groove surfaces, and (3) overheat or otherwise damage the metal metallurgically. After machining, all oil, cutting lubricants, chemicals, dirt, grinding deposits, etc. shall be cleaned off the parts.

3.4. Root Opening. The root openings (G), shown in Figure 1, are maximum values. The actual opening used should be that required to obtain full weld penetration and joint fusion. When the root opening is delineated on the drawing, its value shall be used for manufacture of the components.

3.5 Quality Requirements. The welding grooves shall be within dimensional tolerances shown on the engineering drawings. They shall have reasonably good surfaces consistent with good machining practices. Sharp corners shall be broken a minimum amount for safety only. Correction of machining errors by welding, or any other process of adding material, must be approved by SPPS Materials and Processes engineering.

3.6. Responsibility for Use. The use of the groove designs in this specification, or those delineated on the engineering drawing, does not relieve the manufacturer of components and facilities of the responsibility for producing weld joints to the quality requirements of the applicable welding specification.

4. QUALITY ASSURANCE PROVISIONS

4.1. Inspection. The edge preparations shall be inspected for dimensional requirements as shown in the engineering drawing.

5. PREPARATION FOR DELIVERY. Not applicable.

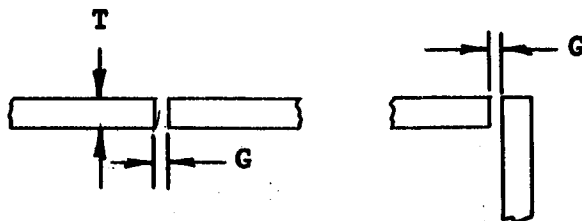
6. DEFINITIONS

6.1. Materials and Processes Engineering. SPPS materials engineer covering the applicable component.

6.2. Design Engineering. SPPS engineer responsible for the design of the applicable component.

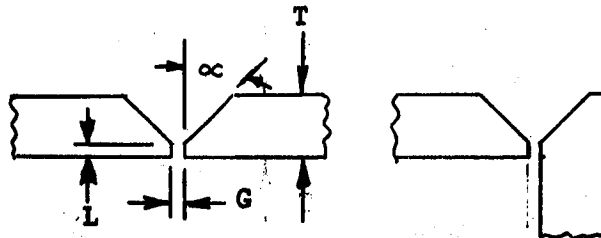
6.3. Applicable Welding Specification. SPPS specification covering the welding of the particular material of which the component is made.

FIGURE 1. WELD JOINT GROOVE DESIGNS

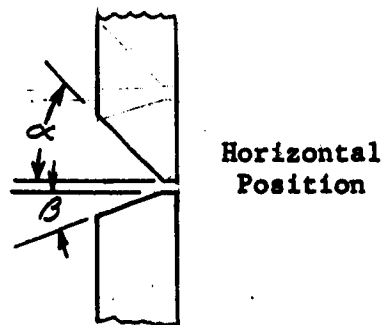


	Dimensions (inch, degree)		
	SS	L-605	Cb, Ta
T max	.13	.095	.06
G max	.06	.05	.03

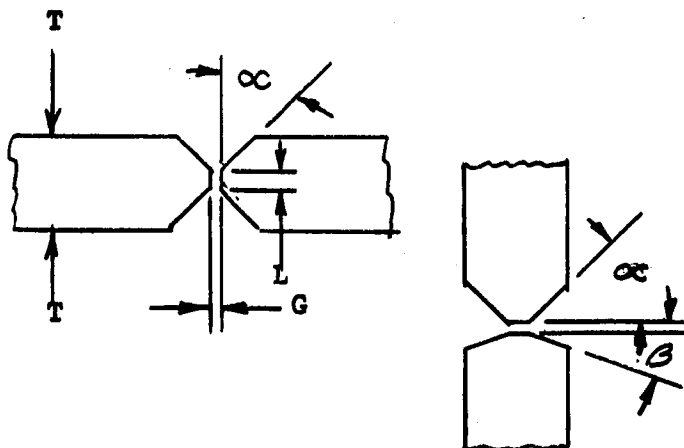
a. Square Groove (one or both sides)



T min	.09	.09	.06
T max	.50	.28	.19
G max	.06	.06	.04
L	.05-.06	.04-.05	.03-.04
α	40	45	45
β	15	15	20



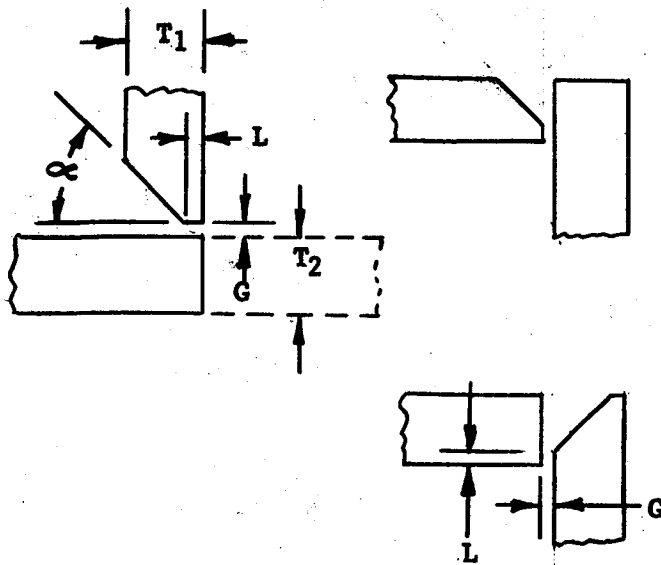
b. Single Vee (one or both sides)



T min	.38	.25	.19
T max	1.0	.75	.50
G max	.06	.06	.04
L	.05-.07	.05-.06	.04-.05
α	40	45	45
β	15	15	20

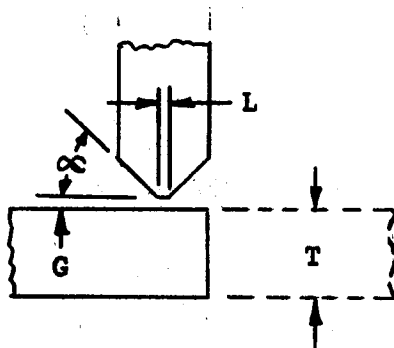
Horizontal Position

c. Double Vee (both sides)



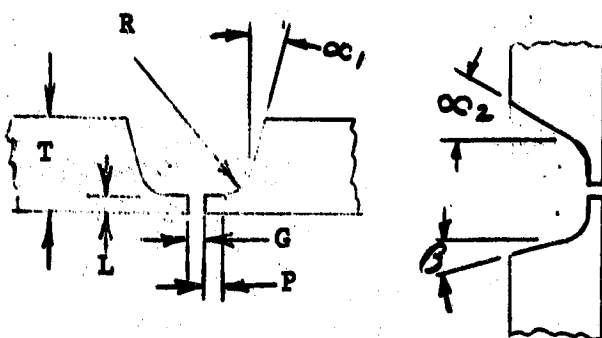
	Dimensions (inch, degree)		
	SS	L-605	Cb, Ta
T ₁ min	.09	.09	.06
T ₁ max	.38	.25	.19
T ₂ min	.09	.09	.06
G max	.09	.06	.06
L	.04-.05	.04-.05	.03-.04
α	45	50	50

d. Single Bevel (one or both sides)



T min	.25	.19	.19
T max	.75	.75	.50
G max	.13	.13	.09
L	.05-.07	.04-.05	.03-.04
α	45	50	50

e. Double Bevel (both sides)

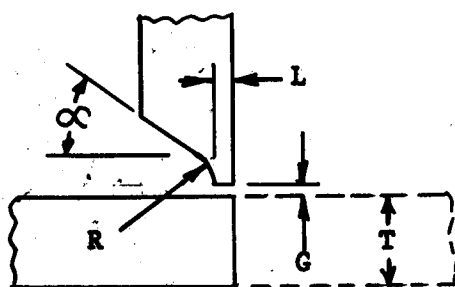


T min	.50	.28	.19
G max	.06	.06	.04
L	.05-.06	.05-.06	.04-.05
P	.09	.06	.05
R	.19	.13	.13
α ₁	15°	20°	20°
α ₂	30°	30°	30°
φ	15°	15°	15°

Horizontal Position

f. Single U (one or both sides)

FIGURE 1. WELD JOINT GROOVE DESIGNS - continued



T min
G max
L
R
 α

Dimensions (inch, degree)		
SS	L-605	Cb, Ta
.44	.31	.22
.09	.06	.06
.05-.06	.05-.06	.04-.05
.19	.16	.12
40°	40°	40°

g. Single J (one or both sides)

03-0016-00-A
SPPS-53
14 August 1964
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SPECIFICATION

STAINLESS STEEL CONOSEAL TUBE UNIONS

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SPECIFICATION

STAINLESS STEEL CONOSEAL TUBE UNIONS

1. SCOPE

1.1. Scope. This specification covers the installation, connection, and leak-checking of Conoseal tube unions to be used in alkali metal handling systems.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents

SPPS-42	Alkali Metal Handling and Control
SPPS-41 (1 May 1964)	Welding of Austenitic Stainless Steels
Bulletin No. 804	Conoseal Union Fitting - Aeroquip Corporation, Marman Division, Los Angeles, California

3. REQUIREMENTS

3.1. Installation Procedure

3.1.1. Tube Welding. The Conoseal tube union fitting is designed for use with standard tube sizes ranging from 1/8-inch outside diameter to one-inch outside diameter. Referring to Aeroquip Bulletin No. 804 and/or Figure 1, both male and female flanges have tube sockets specifically designed for welding the tubing to the flanges. The welding procedure is:

- a. Insert the tubing into the flange socket.
- b. Fuse the tubing to the flange from the inside of the flange in accordance with SPPS-41, taking care not to strike the arc on the flange sealing face. Weld tubing to both flanges in this manner.

c. Remove the oxide film from the flange faces and welds with 20% HCl solution as evidenced by the disappearance of the color from the heat affected area for all unions contacting alkali metal.

3.1.2. Pipe Welding. When using the Conoseal tube union fitting with pipe, it may not be possible to insert the pipe into the tube socket. Therefore, the pipe may be butt-welded to the flanges in accordance with SPPS-41. This type of connection may be used in vacuum or gas lines only--NOT in lines to contain liquid metals.

3.2. Connection Procedure

3.2.1. Connecting. Inspect the gasket and both flange faces to determine that there are no visible burrs or nicks on the sealing surfaces. Insert the gasket into either flange and tighten the union to 500 ± 100 inch-pounds torque.

Re-use of a given union requires replacement of the gasket.

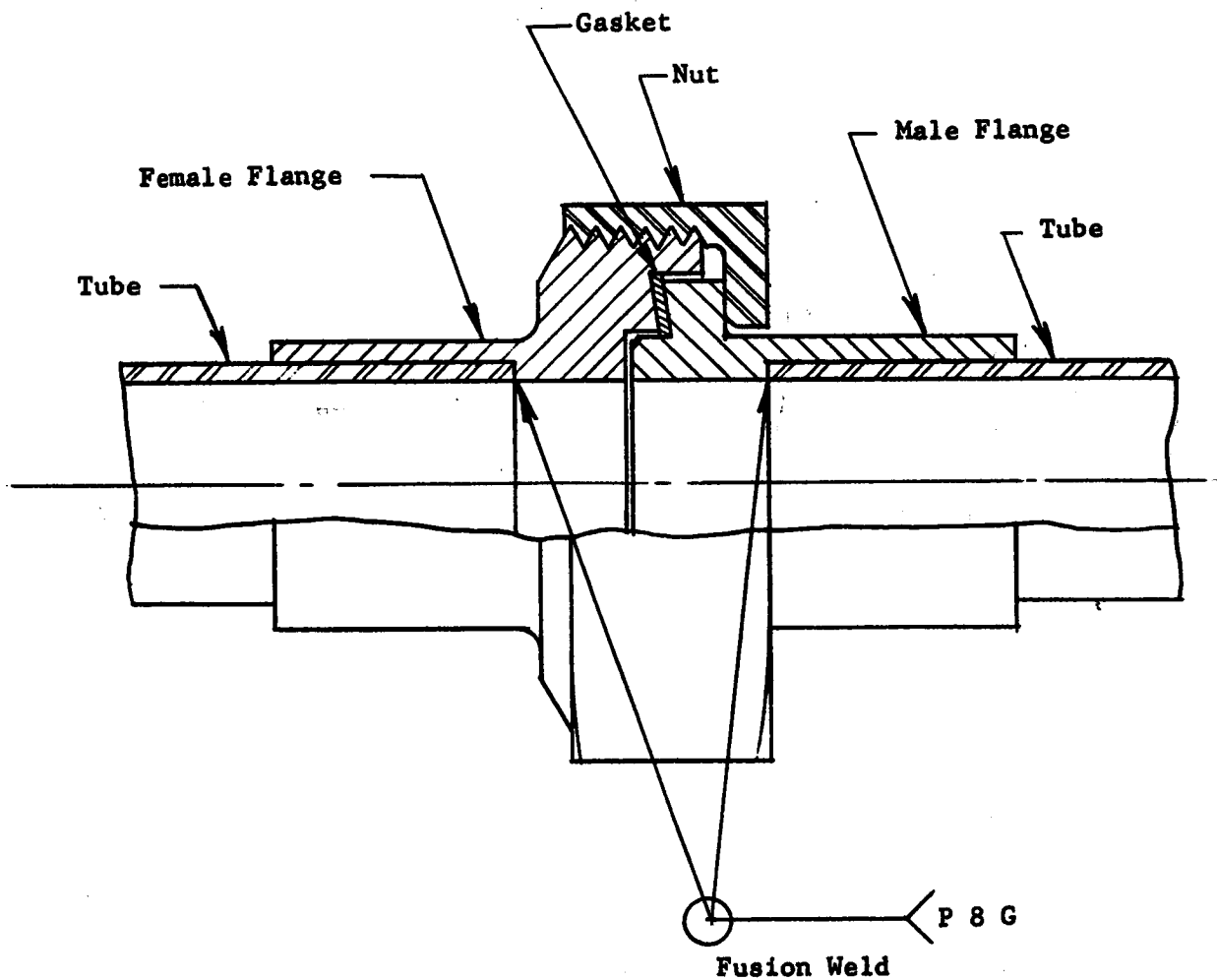
3.3. Leak-Checking Procedure

3.3.1. Leak-Checking. Conoseal unions, after being connected in lines which will serve as either alkali metal transfer lines or gas-vacuum lines, should be helium leak-checked at a sensitivity of 5×10^{-11} std. cc/sec. with a maximum allowable leakage of 5×10^{-10} std. cc/sec. Leak-checking should be done at room temperature, at the anticipated service temperature, and after temperature cycling where applicable.

3.4. Chemical Composition. Referring to Aeroquip Bulletin No. 804, the flanges and gaskets will be made from Type 347 stainless steel and the nut from black iron.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. Compliance with the leakage standards will be recorded in the bound notebook which includes the operating data for a particular program or system. Lack of compliance will also be recorded and corrective action taken; e.g., replacing or tightening the Conoseal union fitting.



NOTE: Use 400 to 600 inch-pounds of torque to tighten nut.

Figure 1. Assembly of Conoseal Union Fitting

APPENDIX E

ALKALI METAL PROCUREMENT AND PURIFICATION SPECIFICATIONS

01-0031-00-B
SPPS-45-I
10 May 1965
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SPECIFICATION

REACTOR GRADE SODIUM METAL

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

Reactor Grade Sodium Metal

- CONTINUED

DATE

10 May 1965

NO.

01-0031-00-B

1. SCOPE

1.1. Scope. This specification covers the reactor grade of high purity sodium metal.

2. APPLICABLE DOCUMENTS

2.1.1. Government Documents

MIL-STD-271C

Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206.

2.2. Non-Government Documents. None.

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgements.

3.2. Chemical Composition

3.2.1. Product Composition. The manufacturers analysis on the product shall conform to the requirements in Table I.

3.2.2. Check Analysis. On receipt, the product will be sampled and re-analyzed, and shown to conform to the requirements in Table II. Non-conformance will be reason for rejection of the shipments.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certifications. The analysis made by the material manufacturers to determine the percentage of elements stipulated in this specification shall be reported to the purchaser at the time of shipment in a certificate of test. The certification will be supplied in triplicate.

4.1.2. Methods of Analysis

4.1.2.1. Vendor Analyses. The chemical analyses shall be performed in accordance with ASTM methods when available. When ASTM methods are not available, the analyses will be performed according to written procedures which result in data of known precision and accuracy. Such procedures will be supplied to the purchaser and the vendor will reference such procedures on test certificates. If procedures are proprietary, the vendor will not supply procedures to the purchaser, but will keep them on file for review with the government contractor, if requested.

SP 1073 A

Reactor Grade Sodium Metal - CONTINUED	DATE 10 May 1965	NO. 01-0031-00-B
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4.1.2.2. Check Analyses. Check analyses will be performed by the purchaser and/or by Nuclear Materials and Equipment Corporation, Apollo, Pa.

4.2. Transfer Methods

4.2.1. Environmental Conditions. Shipping containers will be mass spectrometer lead-checked per MIL-STD-271C. The sodium will be transferred from storage to shipping containers through stainless steel lines and valves under conditions which do not result in an oxygen contamination of greater than 5 ppm. The sodium must not contact brazed or soldered connections or joints during the transfer operation. The maximum transfer temperature will be 250°F. Containers are to be pressurized to 5 psig argon pressure after the sodium is frozen.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each shipping container shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order number, lot number and gross, net and tare weights.

5.2. Packing. Material will be shipped in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206. Each shipment will be legibly and conspicuously marked, "Danger - Hazardous Materials - Keep Away From Water."

6. NOTES. None

Reactor Grade Sodium Metal - CONTINUED

DATE

10 May 1965

NO.

01-0031-00-B

TABLE IPURITY SPECIFICATION FOR REACTOR GRADE SODIUM

<u>Element</u>	<u>Maximum Content</u> (ppm)
Oxygen	50 (in NA) as Na_2O
Chlorine	10 (in Na)
Carbon	20 (in NA)
Potassium	200 (in NaCl)
Tin	2 " "
Lead	10 " "
Cobalt	0.5 " "
Nickel	10 " "
Iron	10 " "
Calcium	10 " "
Lithium	5 " "
Boron	1 " "
Manganese	10 " "
Cadmium	2 " "
Rhodium	30 " "
Indium	25 " "
Rhenium	100 " "
Copper	10 " "
Silver	15 " "
Mercury	35 " "
Europium	0.1 " "
Samarium	0.1 " "
Dysprosium	0.1 " "
Gadolinium	0.1 " "
Sodium	99.95% min.

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Reactor Grade Sodium Metal - CONTINUED

DATE

10 May 1965

NO.

01-0031-00-B

TABLE IICHECK ANALYSIS FOR REACTOR GRADE SODIUM

<u>Element</u>	<u>Maximum Content</u> (ppm)
Oxygen	60 (in Na) as Na_2O
Tin	2 (in NaCl)
Lead	10 " "
Cobalt	5 " "
Nickel	10 " "
Iron	10 " "
Calcium	10 " "
Boron	5 " "
Manganese	10 " "
Copper	10 " "
Silver	15 " "
Magnesium	unspecified
Chromium	"
Silicon	"
Titanium	"
Aluminum	"
Vanadium	"
Zirconium	"
Molybdenum	"
Columbium	"
Barium	"

01-0032-00-B
SPPS-45-II
10 May 1965
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SPECIFICATION

HOT TRAPPED REACTOR GRADE SODIUM METAL

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

Hot Trapped Reactor Grade Sodium Metal

- CONTINUED

DATE

10 May 1965

NO.

01-0032-00-B

1. SCOPE

1.1. Scope. This specification covers the hot trapping and purity of reactor grade sodium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

01-0031-00-B
27 March 1965

Reactor Grade Sodium Metal

03-0018-00-A
15 June 1965

Alkali Metal Handling and
Control Procedures

03-0014-00-A
1 May 1964

Welding of Austenitic Stainless
Steel for Liquid Metal Systems

3. REQUIREMENTS

3.1. Hot Trapping Procedure

3.1.1. Outgassing. The alkali metal will be outgassed in the shipping container, as indicated below, prior to transfer to the hot₃ trap. A vacuum system capable of achieving a blank-off pressure of 1×10^{-3} torr will be used. Trapping will be provided between the vacuum pump and the vacuum-inert gas manifold to prevent contamination of the system by back-streaming of pump oil vapors. The inert gases used (helium and argon) will be shown to contain less than 10 molar ppm combined water and oxygen. Referring to Figure 1, the procedure is:

- a. Make sure that all valves are clean to the seats and then connect the vacuum-inert gas manifold as shown in Figure 1. (See Spec. 03-0018-00-A for valve cleaning.)
- b. Heat the shipping container gas valve and the line connecting it to the shipping container to a temperature of 250°F plus or minus 25°F and pressurize the manifold to about 20 psia with inert gas.
- c. Open the shipping container gas valve to blow out any alkali metal which it may contain.
- d. Evacuate the shipping container through the gas valve, heat it to between 450°F and 600°F and monitor the pressure in the manifold.
- e. When the pressure levels off or drops below 1×10^{-2} torr, close the valve at the trap and measure the pressure rise rate. Continue evacuating and checking pressure rise until the rate is less than 10^{-2} torr-liters per minute.

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Hot Trapped Reactor Grade Sodium Metal - CONTINUED	DATE 10 May 1965	NO. 01-0032-00-B
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- f. Cool the shipping container at a rate not exceeding 50°F per hour to a temperature which exceeds the melting point of the alkali metal by not more than 50°F.

3.1.2. Hot Trapping. The hot trap will be built of an austenitic stainless steel and will be capable of withstanding an internal pressure of 50 psig at 1500°F for at least 10,000 hours. It will be lined with titanium or zirconium sheet, not less than 0.01 inch thick, in a manner which prevents liquid metal between the liner and the hot trap body from returning to the interior of the liner when the hot trap is properly oriented. The getter shall be titanium, zirconium or 50% titanium-50% zirconium alloy sheet, turnings or chunks (not sponge), and the alkali metal weight to getter surface ratio shall not exceed 10 grams/square inch. When it is known that the oxygen content of the getter is greater than 10% by weight for titanium or 5% by weight for zirconium, the liner and getter will be replaced.

All valves used will be of austenitic stainless steel except plugs and/or seats which may be Stellite. They will be of the bellows type, sealed with metal gaskets or welded. Valves which contact the alkali metal will be angle pattern and will be oriented so that the bellows is between the hot trap and valve seat with one exception. The exception is the valve on the connecting line between the shipping container and hot trap. Here the bellows should be between the valve seat and the vacuum-inert gas manifold. All valves should be attached so that the plug axis is vertical and that the handle is toward the ground to facilitate cleaning. (See spec. 03-0018-00-A for valve cleaning.)

All welding will be performed according to Spec 03-0014-00-A.

Before use, the hot trap will be outgassed at a temperature exceeding 250°F until the pressure rise rate is less than 1×10^{-3} torr-liter per minute. It will be helium leak-checked at this temperature with a maximum allowable leak rate of 5×10^{-10} std. cm³ of air per second. Hot traps will have a stainless steel filter having a nominal pore size of 5 microns on the dip leg between the hot trap and the valve.

Referring again to Figure 1, the procedure is:

- Heat the connecting line between the shipping container and hot trap, including valves, to 250°F and evacuate through the vacuum-inert gas valve. Helium leak-check the transfer line.
- Pressurize manifold to 20 psia and open the hot trap gas valve to blow down alkali metal which may be in it.
- Evacuate the hot trap and transfer line to less than one torr inert gas pressure and close the manifold valve.
- Pressurize the shipping container to about 20 psia.

Hot Trapped Reactor Grade Sodium Metal - CONTINUED

DATE

10 May 1965

NO.

01-0032-00-B

- e. Heat all components to a temperature exceeding the melting point of the alkali metal. The temperature of the shipping container filter should not exceed the melting point of the alkali metal by more than 50°F.
- f. Open the gas valve of the hot trap and then the dip leg valve of the shipping container and transfer the desired quantity of metal to the hot trap. (See Spec. 03-0018-00-A for methods for determining quantities transferred.)
- g. Close the dip leg valve on the shipping container and the gas valve on the hot trap. Cool the transfer system to room temperature and remove it, and clean valves.
- h. Reconnect the vacuum-inert gas valve to the hot trap gas valve and evacuate back to the seat of the gas valve.
- i. Heat the gas valve to a temperature exceeding the melting point of the alkali metal and pressurize the manifold to about 17 psia.
- j. Open the hot trap gas valve to pressurize the hot trap to about 17 psia and maintain this pressure during subsequent operations.
- k. Heat the hot trap to a temperature between 1300°F and 1450°F and maintain it at temperature for at least 20 hours.
- l. Cool to about 400°F and remove specimens for analysis. The specimen collecting technique is not specified, but one of a number of methods described in Spec. 03-0018-00-A must be used, depending on the analytical apparatus which applies.

3.2. Chemical Composition

3.2.1. Oxygen. The oxygen content shall be less than 20 ppm by weight as Na₂O in sodium.

3.2.2. Metal Impurities. The metallic impurity levels shall conform to the requirements in Table I.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of the analyses, a report will be prepared in quadruplicate indicating the lot number of the purified batch and referencing the notebook and page numbers covering the purification and analyses. This report will be distributed as follows:

1. Project Engineer in charge of facility in which the sodium will be used.
2. Quality Assurance
3. Alkali Metal Custodian
4. File

SP 1073 A

Hot Trapped Reactor Grade Sodium Metal - CONTINUED

DATE

10 May 1965

NO.

01-0032-00-B

4.2. Chemical Analysis

4.2.1. Specimen Preparation. Analytical specimens will be prepared in accordance with techniques described in Spec. 03-0018-00-A.

4.2.2. Analytical Methods

4.2.2.1. Oxygen. Oxygen analyses will be performed by the amalgamation methods described in Spec. 03-0018-00-A.

4.2.2.2. Metal Impurities. Analyses will be performed in accordance with methods found in Spec. 03-0018-00-A.

5. PREPARATION FOR DELIVERY. None

6. NOTES. None

Hot Trapped Reactor Grade Sodium Metal - CONTINUED

DATE

10 May 1965

NO.

01-0032-00-B

TABLE IANALYSIS OF HOT TRAPPED REACTOR GRADE SODIUM METAL

<u>ELEMENT</u>	<u>MAXIMUM CONTENT</u> <u>(ppm)</u>
Tin	2 (in NaCl)
Lead	10 " "
Cobalt	5 " "
Nickel	10 " "
Iron	10 " "
Calcium	10 " "
Boron	5 " "
Manganese	10 " "
Copper	10 " "
Silver	15 " "
Magnesium	unspecified
Chromium	"
Silicon	"
Titanium	"
Aluminum	"
Vanadium	"
Zirconium	"
Molybdenum	"
Columbium	"
Barium	"

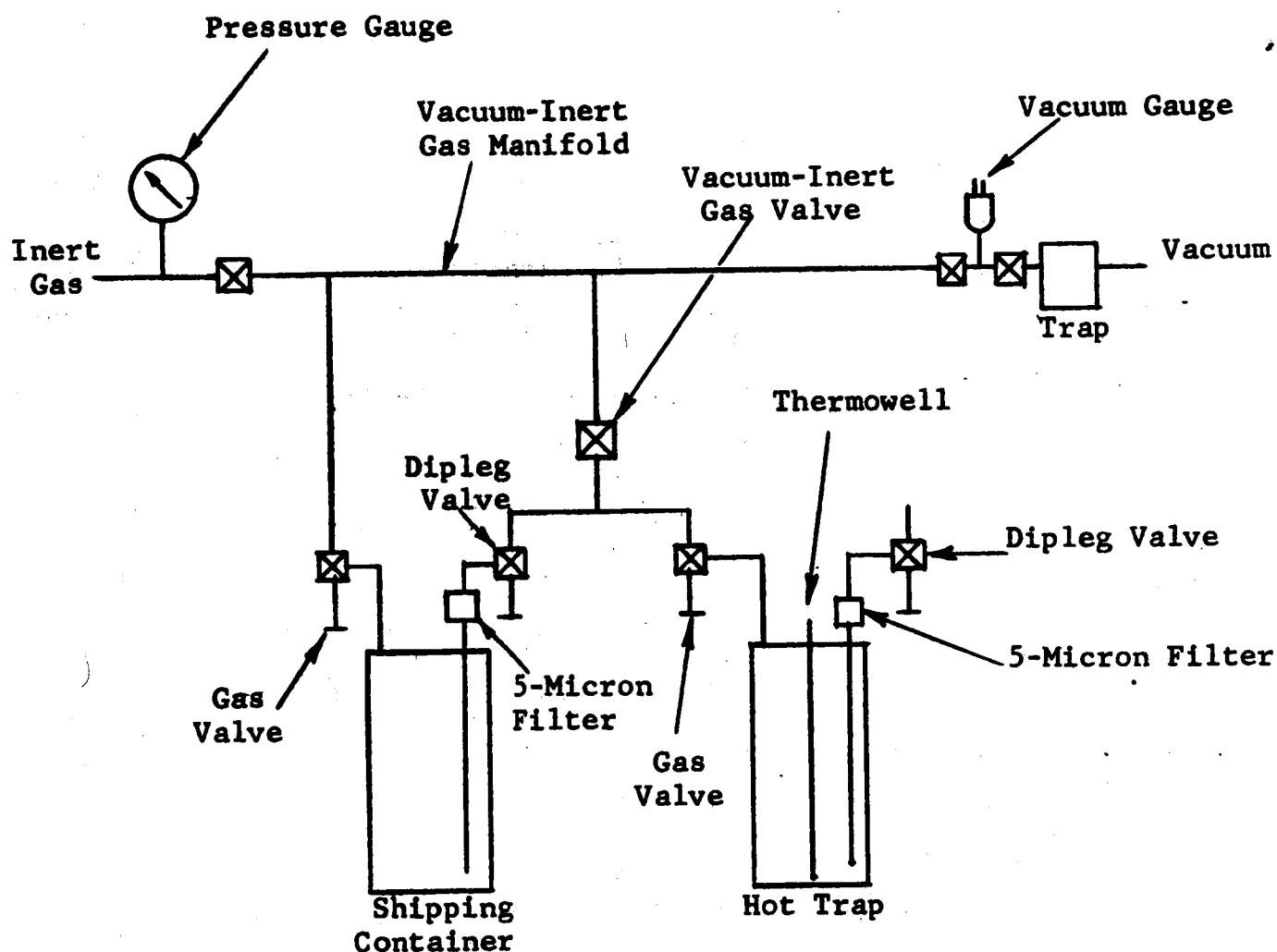


Figure 1. Schematic Drawing of the Apparatus for Outgassing and Hot Trapping Sodium

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10 May 1965
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SPECIFICATION

HIGH PURITY GRADE POTASSIUM METAL

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO 45215

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High Purity Grade Potassium Metal

- CONTINUED

DATE

10 May 1965

NO.

01-0033-00-B

1. SCOPE

1.1. Scope. This specification covers the high purity grade potassium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL - STD - 271C

Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206.

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase acknowledgments.

3.2. Chemical Composition

3.2.1. Product Composition. The manufacturers analysis on the product shall conform to the requirements in Table I.

3.2.2. Check Analysis. On receipt, the product will be sampled and reanalyzed, and shown to conform to the requirements in Table II. Nonconformance will be reason for rejection of the shipments.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certifications. The analysis made by the material manufacturers to determine the percentage of elements stipulated in this specification shall be reported to the purchaser at the time of shipment in a certificate of test. The certification will be supplied in triplicate.

4.1.2. Vendor Analyses. The chemical analyses shall be performed in accordance with ASTM methods when available. When ASTM methods are not available, the analyses will be performed according to written procedures which result in data of known precision and accuracy. Such procedures will be supplied to the purchaser and the vendor will reference such procedures on test certificates. If procedures are proprietary, the vendor will keep them on file for review with the government contractor, if requested.

4.1.2.2. Check Analyses. Check analyses will be performed by the purchaser and/or by Nuclear Materials and Equipment Corporation, Apollo, Pa.

4.2. Transfer Methods

High Purity Grade Potassium Metal

- CONTINUED

DATE

10 May 1965

NO.

01-0033-00-B

4.2.1. Environmental Conditions. Shipping containers will be mass spectrometer leak-checked per MIL-STD-271C. The potassium will be transferred from storage to shipping containers through stainless steel lines and valves under conditons which do not result in an oxygen contamination of greater than 5 ppm. The potassium must not contact brazed or soldered connections or joints during the transfer operation. The maximum transfer temperature will be 250°F. Containers are to be pressurized to 5 psig argon pressure after the potassium is frozen.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each shipping container shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order number, lot number and gross, net and tare weights.

5.2. Packing. Material will be shipped in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206. Each shipment will be legibly and conspicuously marked "Danger - Hazardous Materials - Keep Away from Water."

6. NOTES. None

High Purity Grade Potassium Metal

- CONTINUED

DATE

10 May 1965

NO.

01-0033-00-B

TABLE IPURITY SPECIFICATION FOR HIGH GRADE POTASSIUM

<u>Element</u>	<u>Maximum Content</u> (ppm)
Oxygen	50 (in K) as K_2O
Sodium	50 (in KCl)
Tin	5 "
Lead	5 "
Cobalt	5 "
Nickel	25 "
Iron	50 "
Boron	10 "
Manganese	10 "
Copper	30 "
Silver	5 "
Aluminum	10 "
Magnesium	8 "
Chromium	5 "
Silicon	25 "
Titanium	5 "
Molybdenum	3 "
Vanadium	5 "
Beryllium	5 "
Zirconium	10 "
Strontium	5 "
Barium	5 "
Calcium	25 "
Potassium	99.99% min.

High Purity Grade Potassium Metal

- CONTINUED

DATE

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NO.

01-0033-00-B

TABLE IICHECK ANALYSIS FOR HIGH PURITY GRADE POTASSIUM

<u>Element</u>	<u>Maximum Content</u> (ppm)
Oxygen	60 (in K) as K_2O
Sodium	50 (in KCl)
Tin	5 "
Lead	5 "
Cobalt	5 "
Nickel	25 "
Iron	50 "
Calcium	25 "
Manganese	10 "
Copper	30 "
Silver	5 "
Magnesium	8 "
Chromium	5 "
Silicon	25 "
Titanium	5 "
Aluminum	10 "
Zirconium	10 "
Molybdenum	5 "
Columbium	5 "

01-0034-00-B

SPPS-46-II

10 May 1965

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SPECIFICATION

HOT TRAPPED HIGH PURITY GRADE POTASSIUM METAL

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

SP 1073 A

Hot Trapped High Purity Grade Potassium Metal- CONTINUED

DATE

10 May 1965

NO.

01-0034-00-B

1. SCOPE

1.1. Scope. This specification covers the hot trapping and purity of high purity grade potassium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

01-0033-00-B

10 May 1965

High Purity Grade Potassium Metal

03-0018-00-A

15 June 1965

Alkali Metal Handling and Control Procedures

03-0014-00-A

1 May 1964

Welding of Austenitic Stainless Steels for Liquid Metal Systems

3. REQUIREMENTS

3.1. Hot Trapping Procedure

3.1.1. Outgassing. The alkali metal will be outgassed in the shipping container, as indicated below, prior to transfer to the hot trap. A vacuum system capable of achieving a blank-off pressure of 1×10^{-3} torr will be used. Trapping will be provided between the vacuum pump and the vacuum-inert gas manifold to prevent contamination of the system by back-streaming of pump oil vapors. The inert gases used (helium or argon) will be shown to contain less than 10 molar ppm combined water and oxygen. Referring to Figure 1, the procedure is:

- a. Make sure that all valves are clean to the seats and then connect the vacuum-inert gas manifold as shown in Figure 1. (See Spec. 03-0018-00-A for valve cleaning.)
- b. Heat the shipping container gas valve and the line connecting it to the shipping container to a temperature of 250°F plus or minus 25°F and pressurize the manifold to about 20 psia with inert gas.
- c. Open the shipping container gas valve to blow down any alkali metal which it may contain.
- d. Evacuate the shipping container through the gas valve, heat it to between 450°F and 600°F and monitor the pressure in the manifold.
- e. When the pressure levels off or drops below 1×10^{-2} torr, close the valve at the trap and measure the pressure rise rate. Continue evacuating and checking pressure rise until the rate is less than 10^{-2} torr-liters per minute.

SP 1073 A

Hot Trapped High Purity Grade Potassium Metal- CONTINUED

DATE

10 May 1965

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01-0034-00-B

- f. Cool the shipping container at a rate not exceeding 50°F per hour to a temperature which exceeds the melting point of the alkali metal by not more than 50°F .

3.1.2. Hot Trapping. The hot trap will be built of an austenitic stainless steel and will be capable of withstanding an internal pressure of 50 psig at 1500°F for at least 10,000 hours. It will be lined with titanium or zirconium sheet, not less than 0.01 inch thick, in a manner which prevents liquid metal between the liner and the hot trap body from returning to the interior of the liner when the hot trap is properly oriented. The getter shall be titanium, zirconium or 50% titanium-50% zirconium alloy sheet, turnings or chunks (not sponge), and the alkali metal weight to getter surface ratio shall not exceed 10 grams/square inch. When it is known that the oxygen content of the getter is greater than 10% by weight for titanium of 5% by weight for zirconium, the liner and getter will be replaced.

All valves used will be of austenitic stainless steel except plugs and/or seats which may be Stellite. They will be of the bellows type, sealed with metal gaskets or welded. Valves which contact the alkali metal will be angle pattern and will be oriented so that the bellows is between the hot trap and valve seat with one exception. The exception is the valve on the connecting line between the shipping container and hot trap. Here the bellows should be between the valve seat and the vacuum-inert gas manifold. All valves should be attached so that the plug axis is vertical and that the handle is toward the ground to facilitate cleaning. (See Spec. 03-0018-00-A for valve cleaning.)

All welding will be performed according to Spec. 03-0014-00-A .

Before use, the hot trap will be outgassed at a temperature exceeding 250°F until the pressure rise rate is less than 1×10^{-5} torr-liter per minute. It will be helium leak-checked at this temperature with a maximum allowable leak rate of 5×10^{-10} std. cm³ of air per second. Hot traps will have a stainless steel filter having a nominal pore size of 5 microns on the dip leg between the hot trap and the valve.

Referring again to Figure 1, the procedure is:

- Heat the connecting line between the shipping container and hot trap, including valves, to 250°F and evacuate through the vacuum-inert gas valve. Helium leak-check transfer line.
- Pressurize manifold to 20 psia and open the hot trap gas valve to blow down alkali metal which may be in it.
- Evacuate the hot trap and transfer line to less than one torr inert gas pressure and close the manifold valve.
- Pressurize the shipping container to about 20 psia.

Hot Trapped High Purity Grade Potassium Metal. CONTINUED

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- e. Heat all components to a temperature exceeding the melting point of the alkali metal. The temperature of the shipping container filter should not exceed the melting point of the alkali metal by more than 50 F.
- f. Open the gas valve of the hot trap and then the dip leg valve of the shipping container and transfer the desired quantity of metal to the hot trap. (See Spec. 03-0018-00-A for methods for determining quantities transferred.)
- g. Close the dip leg valve on the shipping container and the gas valve on the hot trap. Cool the transfer system to room temperature and remove it, and clean valves.
- h. Reconnect the vacuum-inert gas valve to the hot trap gas valve and evacuate back to the seat of the gas valve.
- i. Heat the gas valve to a temperature exceeding the melting point of the alkali metal and pressurize the manifold to about 17 psia.
- j. Open the hot trap gas valve to pressurize the hot trap to about 17 psia and maintain this pressure during subsequent operations.
- k. Heat the hot trap to a temperature between 1300°F and 1450°F and maintain it at temperature for at least 20 hours.
- l. Cool to about 400°F and remove specimens for analysis. The specimen collecting technique is not specified, but one of a number of methods described in Spec. 03-0018-00-A must be used, depending on the analytical apparatus which applies.

3.2. Chemical Composition

3.2.1. Oxygen. The oxygen content shall be less than 20 ppm by weight as K₂O in sodium.

3.2.2. Metal Impurities. The metallic impurity levels shall conform to the requirements in Table I.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of the analyses, a report will be prepared in quadruplicate indicating the lot number of the purified batch and referencing the notebook and page number covering the purification and analyses. This report will be distributed as follows:

1. Project Engineer in charge at facility in which the potassium will be used.
2. Quality Assurance Engineer
3. Alkali Metal Custodian
4. File

Hot Trapped High Purity Grade Potassium Metal- CONTINUED	DATE 10 May 1965	NO. 01-0034-00-B
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4.2. Chemical Analysis

4.2.1. Specimen Preparation. Analytical specimens will be prepared in accordance with techniques described in Spec. 03-0018-00-A.

4.2.2. Analytical Methods

4.2.2.1. Oxygen. Oxygen analyses will be performed by the methods described in Spec. 03-0018-00-A.

4.2.2.2. Metal Impurities. Analyses will be performed in accordance with methods found in Spec. 03-0018-00-A.

5. PREPARATION FOR DELIVERY. None

6. NOTES. None.

Hot Trapped High Purity Grade Potassium Metal- CONTINUED

DATE

10 May 1965

NO.

01-0034-00-B

TABLE IANALYSIS OF HOT TRAPPED HIGH PURITY GRADE POTASSIUM METAL

<u>Element</u>	<u>Maximum Content</u> <u>(ppm)</u>
Tin	5 (in KCl)
Lead	5 " "
Cobalt	5 " "
Nickel	10 " "
Iron	10 " "
Calcium	25 " "
Manganese	10 " "
Copper	30 " "
Silver	5 " "
Magnesium	8 " "
Chromium	5 " "
Silicon	25 " "
Titanium	5 " "
Aluminum	10 " "
Zirconium	10 " "
Molybdenum	5 " "
Columbium	5 " "

SP 1073 A

HOT TRAPPED HIGH PURITY GRADE
POTASSIUM METAL

- CONTINUED

DATE

5/10/65

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01-0034-00-B

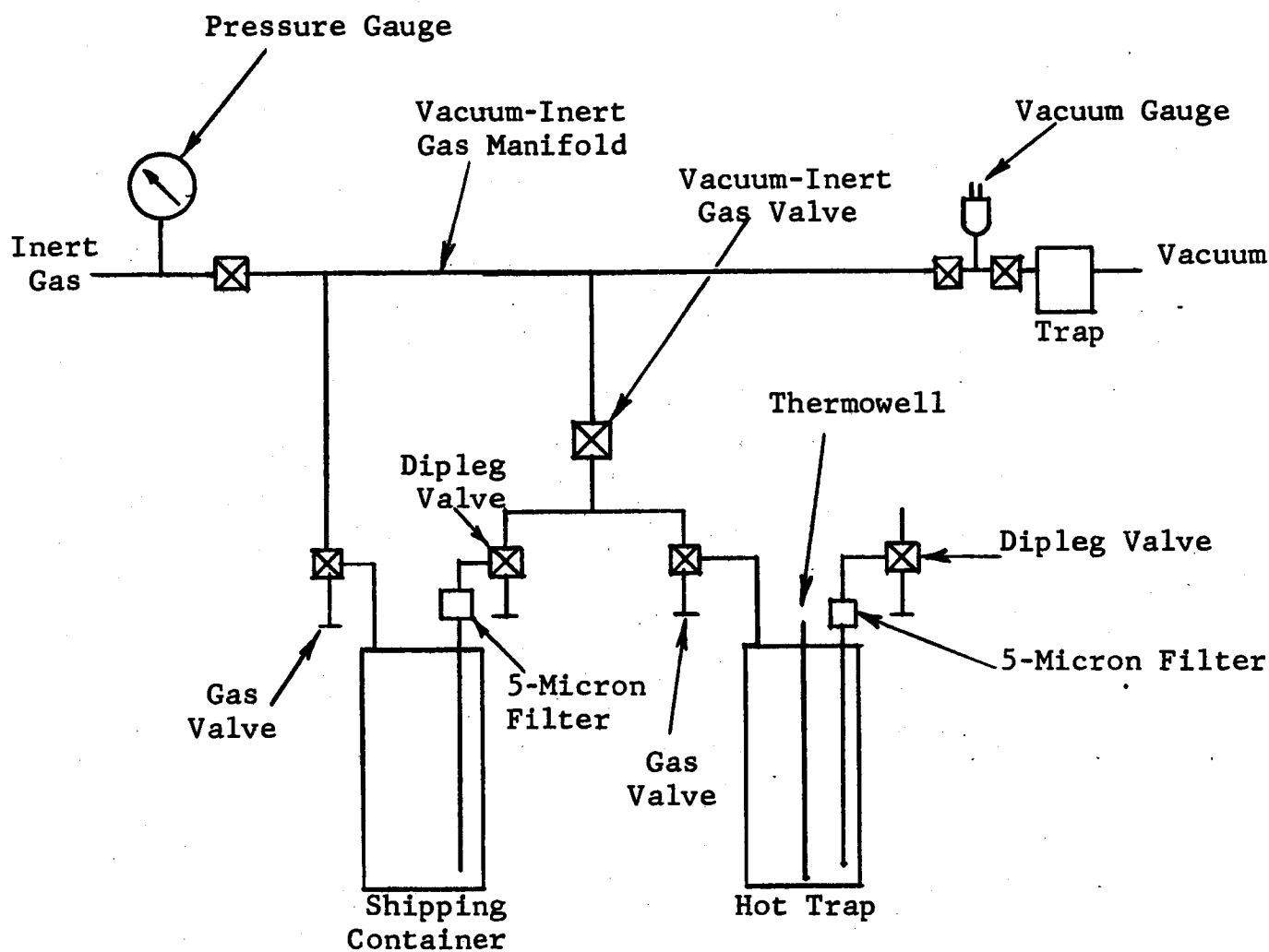


Figure 1. Schematic Drawing of the Apparatus for Outgassing and Hot Trapping Potassium

SPECIFICATION

ALKALI METAL HANDLING AND CONTROL PROCEDURES

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC COMPANY

CINCINNATI, OHIO 45215

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED

DATE

15 June 1965

NO.

03-0018-00-A

1. SCOPE

1.1. Scope. This specification covers all technical phases of the procurement, surveillance, transfer, storage, purification, sampling, analysis, monitoring, usage and handling of alkali metals.

2. APPLICABLE DOCUMENTS2.1. Government Documents

MIL-STD-271C (Ships)
(1 October 1963)

Non-Destructive Testing
Requirements for Metals

Interstate Commerce Commission
Regulations Applying to
Shipping, Part 73-206

2.2. Non-Government Documents

03-0013-00-A
15 September 1964

Mass Spectrometer Leak Detection
Using Helium

03-0014-00-A
1 May 1964

Welding of Austenitic Stainless
Steels

01-0030-00-A
21 January 1965

High Purity Lithium Metal

01-0031-00-B
10 May 1965

Reactor Grade Sodium Metal

01-0032-00-B
10 May 1965

Hot Trapped Reactor Grade Sodium
Metal

01-0033-00-B
10 May 1965

High Purity Grade Potassium
Metal

01-0034-00-B
10 May 1965

Hot Trapped High Purity Grade
Potassium Metal

01-0049-00-A
Not yet written

High Purity Grade Eutectic NaK

01-0050-00-A
Not yet written

Hot Trapped High Purity Grade
Eutectic NaK

01-0051-00-A
Not yet written

High Purity Grade Rubidium
Metal

01-0052-00-A
Not yet written

High Purity Grade Cesium Metal

03-0016-00-A
9 August 1964

Stainless Steel Conoseal Tube
Unions

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED

DATE

15 June 1965

NO.

03-0018-00-A

3. REQUIREMENTS3.1. Procurement

3.1.1. Work Authorizations. Work Authorizations (Form GT 3099-A(4-62)) for the purchase of alkali metals will be initiated by personnel responsible for the operation of a given facility or by the alkali metal custodian when requested to write the Work Authorization by operating personnel. When a Work Authorization is initiated for the purchase of an alkali metal, it will be accompanied by an Alkali Metal Surveillance Record (Form SP 1061), an example of which is attached to this specification as Appendix A. The correct use of this record is explained in paragraph 3.2. of this specification.

3.1.2. Procurement Specifications. Alkali metals will be purchased in accordance with the appropriate specification. A listing of these specifications is given in paragraph 2.2. of this specification.

3.1.3. Approvals. No alkali metal will be purchased without the approval of the Alkali Metal Custodian. Approval for purchase will be indicated when both the Materials Specification and the Purification Specification indicated on the Alkali Metal Surveillance Record have been approved by the Alkali Metal Custodian.

3.2. Surveillance

3.2.1. Purpose. The purpose of the surveillance function is to provide continuous information concerning all alkali metals in the plant. It will yield immediate knowledge about the quantities, location and history of each lot of metal. Furthermore, it will help ensure that a particular lot of alkali metal is appropriate for the facility in which it is being used. The surveillance function is also designed to provide quality assurance information.

3.2.2. Alkali Metal Surveillance Record. The Alkali Metal Surveillance Record (Form SP 1061) will, when completed, provide the information required to accomplish the task set forth in the preceding paragraph. Referring to the example of this record shown as Appendix A, the method of use is as follows:

a. The requestor will at the time of initiation of a Work Authorization attach a copy of the Surveillance Record after filling in the following items: Requestor, Vendor, W/A Number, Intended Use, Material Specification, and Purification Specification.

b. The requestor will send the W/A and Surveillance Record to the Alkali Metal Custodian.

c. The Alkali Metal Custodian will review the W/A and Surveillance Record and approve the Material Specification and the Purification Specification, or will make necessary changes in the Specifications and then approve. The requestor will be informed of any changes.

d. The W/A will be revised, if necessary, approved by the Custodian and sent to Purchasing. The Surveillance Record will be retained by the Custodian.

e. Purchasing will, after approval by Finance, place the order and inform the Custodian of the order number.

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ALKALI METAL HANDLING AND CONTROL PROCEDURES CONTINUED

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f. On receipt of the material, the Custodian will arrange for its transfer to an approved place of storage or to an analytical facility where the material can be sampled.

g. After sampling, the Custodian will arrange for its transfer to an approved place of storage.

h. After receipt of the vendor's analysis and completion of the "in-house" analyses, the Custodian will review the Surveillance Record and approve the material for use if the material conforms to the Specification. If it does not meet the Specification, the Custodian will arrange to have the material returned to the vendor or will waive that part of the Specification involved and then approve the material for use and indicate why the material may be used despite non-conformance.

i. The Custodian will make the necessary arrangements to release the material from storage and the requestor will arrange for the transfer to the facility.

j. When testing is complete or the material is of no further use, it will be disposed of by burning or hydrolyzing, returned to storage, or transferred to shipping for return to the vendor. The requestor will make these arrangements and inform the Custodian by means of an autogram. The autogram must indicate the Material Designation Number and the planned disposition.

3.3. Transportation.

3.3.1. Interplant Transportation. Transfer of alkali metals by commercial carriers between plants will be in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206.

3.3.2. Intraplant Transportation. Quantities of alkali metals in excess of one pound will be transported in steel, stainless steel, or other suitable metal containers which are sealed against water and atmosphere. Quantities in excess of ten pounds will be transported on fork lift trucks or on the beds of open body trucks. Containers containing less than ten pounds of alkali metal may be transported in a passenger automobile, but only with the approval of the Custodian or Safety Specialist. The only alkali metal which may be transported on any public transportation vehicle such as a plant bus will be analytical specimens weighing less than one pound.

Quantities of alkali metals weighing less than a pound may be transported in glass containers which are sealed against water and the atmosphere. Such containers must be enclosed in a metal container with a tightly fitting lid, such as a paint can. The glass container must be cushioned with glass wool, steel wool, or exploded mica (Vermiculite). Combustibles such as cotton, cloths, wood chips, etc. must not be used as a cushioning agent.

3.4. Storage and Inventories

3.4.1. Approved Storage Areas. At present there is one approved place of permanent storage for alkali metals. It is the storage room of Building 309.

3.4.2. Inventories. The total alkali metal inventory consists of that which is stored in Building 309 and those quantities which are in use or being kept temporarily at various approved locations. These approved locations, the quantities permitted in each, and the conditions governing storage and use are indicated in the following paragraph.

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED

DATE

15 June 1965

NO.

03-0018-00-A

3.4.2.1. Permissible Quantities and Approved Locations. The locations where alkali metals may be used and stored, the quantities permitted in each, and the conditions governing storage and use are determined by consideration of probable damage to equipment. These regulations have been formulated in accordance with the desires of the insurance company involved, and a failure to follow them could result in a considerable increase in insurance rates.

Approved locations and permissible quantities are shown in the table in Appendix B of this specification.

3.4.2.2. Periodic Checking of Inventories. The Alkali Metal Custodian will, at quarterly intervals, take an inventory of all alkali metals. This inventory will verify the Surveillance Records and determine whether or not there are cases of non-conformance with the procedures specified herein.

3.4.3. Containment Vessels. Alkali metals weighing more than one pound must be stored in metal containers, sealed against atmosphere and water; the containers to be fabricated from a metal which is resistant to attack by the contained alkali metal; i.e., steel or stainless steel. Stainless steel packless valves with corrosion resistant bellows or diaphragms should be used. Such containers, excluding vendor shipping containers, should be welded in accordance with 03-0014-00-A and leak-checked, prior to use, in accordance with 03-0013-00-A or MIL-STD-271C (Ships). The requirements for vendor shipping containers will be found in the applicable Materials Specifications.

Glass containers should, in general, be avoided for the storage of alkali metals. However, quantities weighing less than one pound may be stored in glass containers which are sealed against the atmosphere and water. Such containers must be packed carefully in a cushioning material such as glass wool, steel wool or Vermiculite inside of a steel container which has a tightly fitting lid, such as a paint can.

All supply containers (such as shipping containers) must be labelled. The label should indicate the alkali metal, the Material Designation Number which appears on the Surveillance Record, the original requestor, the tare of the container, the amount of alkali metal remaining, the name of the person who determined the amount remaining, and the date of the determination. Such labels must be protected so that they are not defaced during handling and storage.

3.5. Purification. Purification procedures will differ for different alkali metals and for different facilities. Consequently, the approved purification procedures have been written up as separate specifications. These specifications are listed in paragraph 2.2. of this specification; for example, 01-0034-00-B gives the approved procedure for hot trapping potassium.

3.5.1. Waiver of Purification Specifications. Under certain circumstances it may be possible to waive a certain purification specification or a portion thereof or to modify it. Such changes can be made only with the approval of the Alkali Metal Custodian, and must be documented on the Surveillance Record before the material is approved for use.

DATE

NO.

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED

15 June 1965

03-0018-00-A

3.6. Chemical Analytical Methods3.6.1. Methods for Determining Oxygen in Alkali Metals3.6.1.1. The Amalgamation Method for Oxygen In Potassium, Sodium and NaKTable of Contents3.6.1.1.1. Introduction3.6.1.1.2. Mercury Purification3.6.1.1.2.1. Cleaning Procedure3.6.1.1.2.1.1. Preliminary Cleanup3.6.1.1.2.1.2. Oxide Removal3.6.1.1.3. Amalgamation Analytical Apparatus3.6.1.1.3.1. Vacuum System3.6.1.1.3.2. Inert Gas Purification System3.6.1.1.4. Equipment Preparation3.6.1.1.4.1. Extrusion Sampling3.6.1.1.4.1.1. Extruder Sample Preparation3.6.1.1.4.1.2. Cutter Preparation3.6.1.1.4.1.3. Flask Preparation3.6.1.1.4.1.4. Stirring Bar Preparation3.6.1.1.4.1.5. Waste Boat Preparation3.6.1.1.5. Apparatus Assembly3.6.1.1.5.1. Assembly3.6.1.1.5.2. Outgassing3.6.1.1.5.2.1. Inert Gas Operation3.6.1.1.5.2.2. Vacuum Operation3.6.1.1.6. Analytical Procedure3.6.1.1.6.1. Inert Gas Operation3.6.1.1.6.1.1. Extrusion Sampling - Sodium and Potassium

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED

DATE

15 June 1965

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3.6.1.1.6.1.2. Liquid Sampling - Sodium, Potassium and NaK3.6.1.1.6.1.2.1. Liquid Sampling Apparatus3.6.1.1.6.1.2.2. Liquid Sampling Procedure3.6.1.1.6.1.3. Sample Amalgamation3.6.1.1.6.1.4. Extraction of the Alkali Metal from the Mercury3.6.1.1.6.1.5. "Oxide" Residue Removal3.6.1.1.6.1.6. "Oxide" Residue Analysis3.6.1.1.6.1.6.1. Titrimetric Analyses3.6.1.1.6.1.6.1.1. Titration Apparatus3.6.1.1.6.1.6.1.2. Titration Procedure3.6.1.1.6.1.6.1.3. Titration Calculation3.6.1.1.6.1.6.2. Flame Photometric Analysis3.6.1.1.6.1.7. Sample Weight Determination3.6.1.1.6.2. Vacuum Operation

3.6.1.1.1. Introduction. The amalgamation method for oxygen in alkali metals consists of amalgamating the alkali metal with the mercury and allowing the insoluble "oxide" to separate from and float to the top of the amalgam, after which the "oxide" quantity is determined by titrimetric or flame photometric techniques. As with other analytical methods, the results produced by the amalgamation method are influenced by the specific techniques, apparatus and conditions used in employing the method. Consequently, the detailed procedures used in conducting the various operations involved in employing the method, as well as the equipment used, is presented as follows.

3.6.1.1.2. Mercury Purification

3.6.1.1.2.1. Cleaning Procedure. The initial amalgamation reaction involves one to five grams of alkali metal and about 1000 grams of mercury. The mercury, therefore, must be free of reducible oxides as well as sodium or potassium oxides if the amalgamation method is to give an accurate indication of the oxygen content of the alkali metal.

3.6.1.1.2.1.1. Preliminary Cleanup. The mercury is washed with hydrochloric acid and then drained through a separatory funnel to remove the acid. It is then washed with water and drained through a separatory funnel to remove the film coating the mercury and the water. This step is repeated until the mercury surface is free of surface film. Acetone is added to the drained mercury to remove the traces of water, using the separatory funnel. The mercury is drained into hexane and then into a dry separatory funnel. This washing process cleans the mercury of alkali metal salts, water, grease and other impurities.

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3.6.1.1.2.1.2. Oxide Removal. The mercury is purified further by oxidizing the other metallic species in the mercury and then mechanically separating these oxides from the mercury by floatation and filtration. The apparatus used for this is a Bethlehem oxifier. The mercury is agitated in a revolving container for four hours with warm air bubbling through the mercury to oxidize the impurities. The mercury-oxide mixture is then allowed to sit in a separatory funnel for at least twenty-four hours during which the oxidized materials float to the top. The pure mercury is then drained from the bottom of the funnel through a gold-adhesion filter into a clean polyethylene bottle and stored for later use.

3.6.1.1.3. Amalgamation Analytical Apparatus. The apparatus used in employing the amalgamation method is shown in Figures 3.6.1.1. and 3.6.1.2. The basic components of the apparatus are:

- a. a stainless steel extruder or a liquid sampling reservoir
- b. an amalgamation reaction flask
- c. a mercury supply reservoir
- d. a stainless steel cutter assembly
- e. a vacuum-argon manifold
- f. a cold trap
- g. a titanium boat to accept the waste sample
- h. a nickel stirring bar and magnet
- i. Viton O-ring seals

A sketch of the extruder is shown in Figure 3.6.1.3. All components are of stainless steel with Viton O-ring seals.

The liquid sampling reservoir and transfer system is shown in Figure 3.6.1.4.

3.6.1.1.3.1. Vacuum System. The vacuum system shown in Figure 3.6.1.1. consists of a Welch Duo-Seal roughing pump and a VEECO model VS-9 pumping system, which includes an air-cooled, oil diffusion pump and optically dense cold trap.

3.6.1.1.3.2. Inert Gas Purification System. The argon or helium used passes through a molecular sieve dryer (Linde type 13X) and a 1500° F hot trap containing titanium chips.

3.6.1.1.4. Equipment Preparation

3.6.1.1.4.1. Extrusion Sampling. An analytical sample of solid sodium or potassium is obtained by extruding the metal into the reaction section with the extruder system shown in Figure 3.6.1.3., and shown attached to the reaction assembly in Figures 3.6.1.1. and 3.6.1.2.

The extruder is designed to accept 0.5-inch outside diameter tubing only. The plunger diameter is 0.437 inch and requires that 20-mil wall tubing be used. The minimum sample tubing length is 12 inches if Swagelok fittings are used to seal the ends.

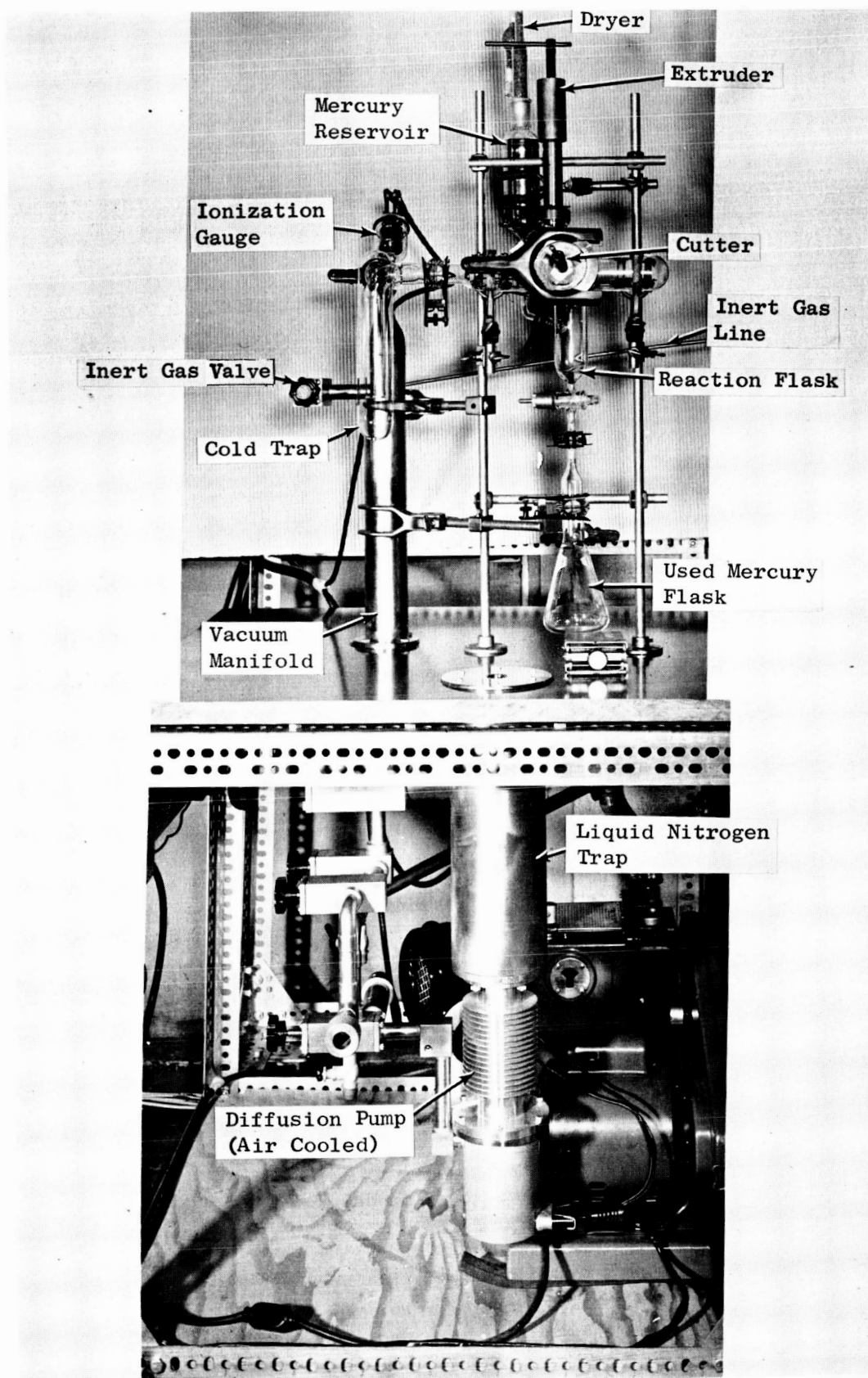


Figure 3.6.1.1. Vacuum-Inert Gas Amalgamation Apparatus Showing Vacuum System. (C64041572), -365-

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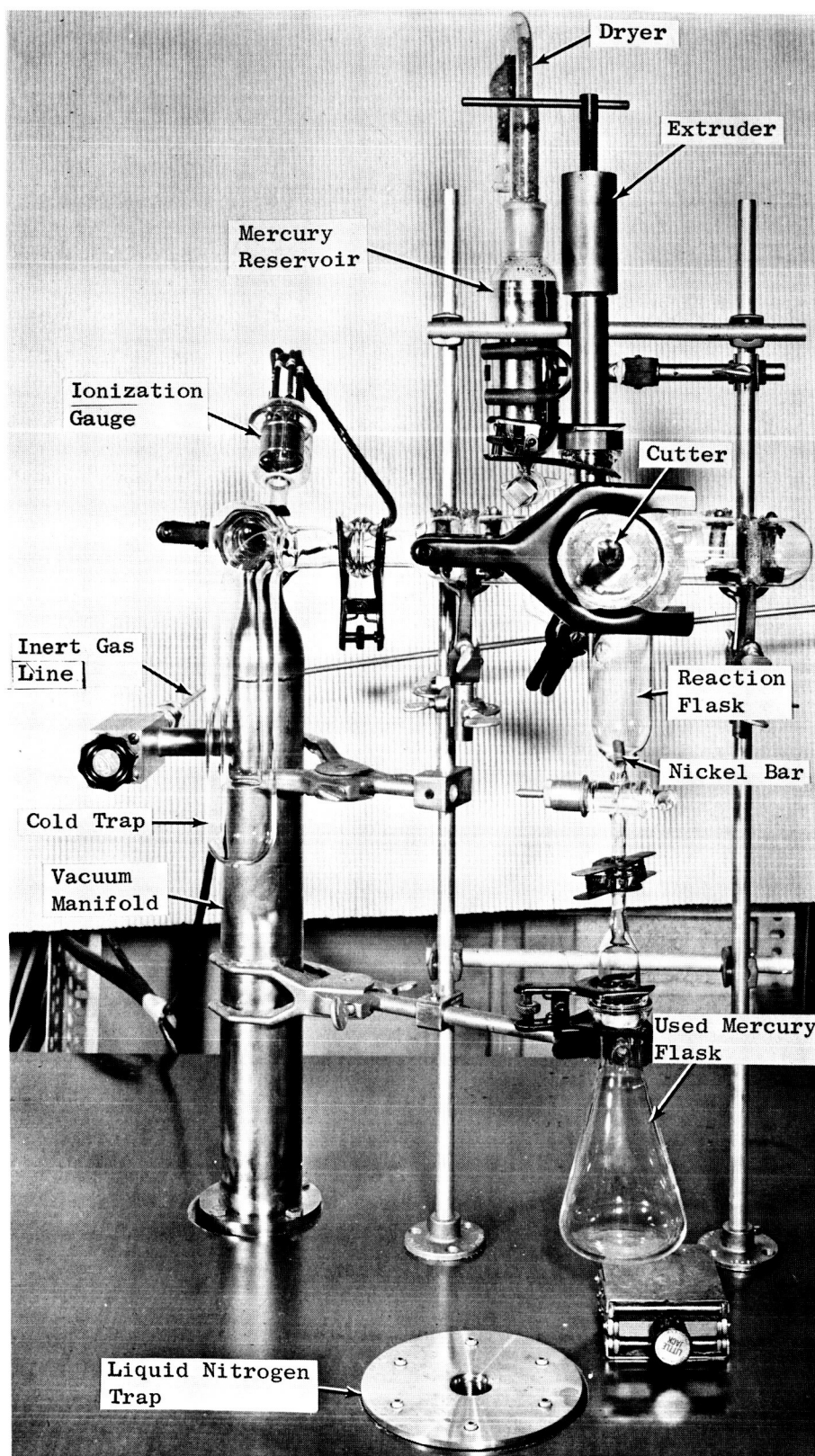


Figure 3.6.1.2. Vacuum-Inert Gas Amalgamation Apparatus. (C64041574).

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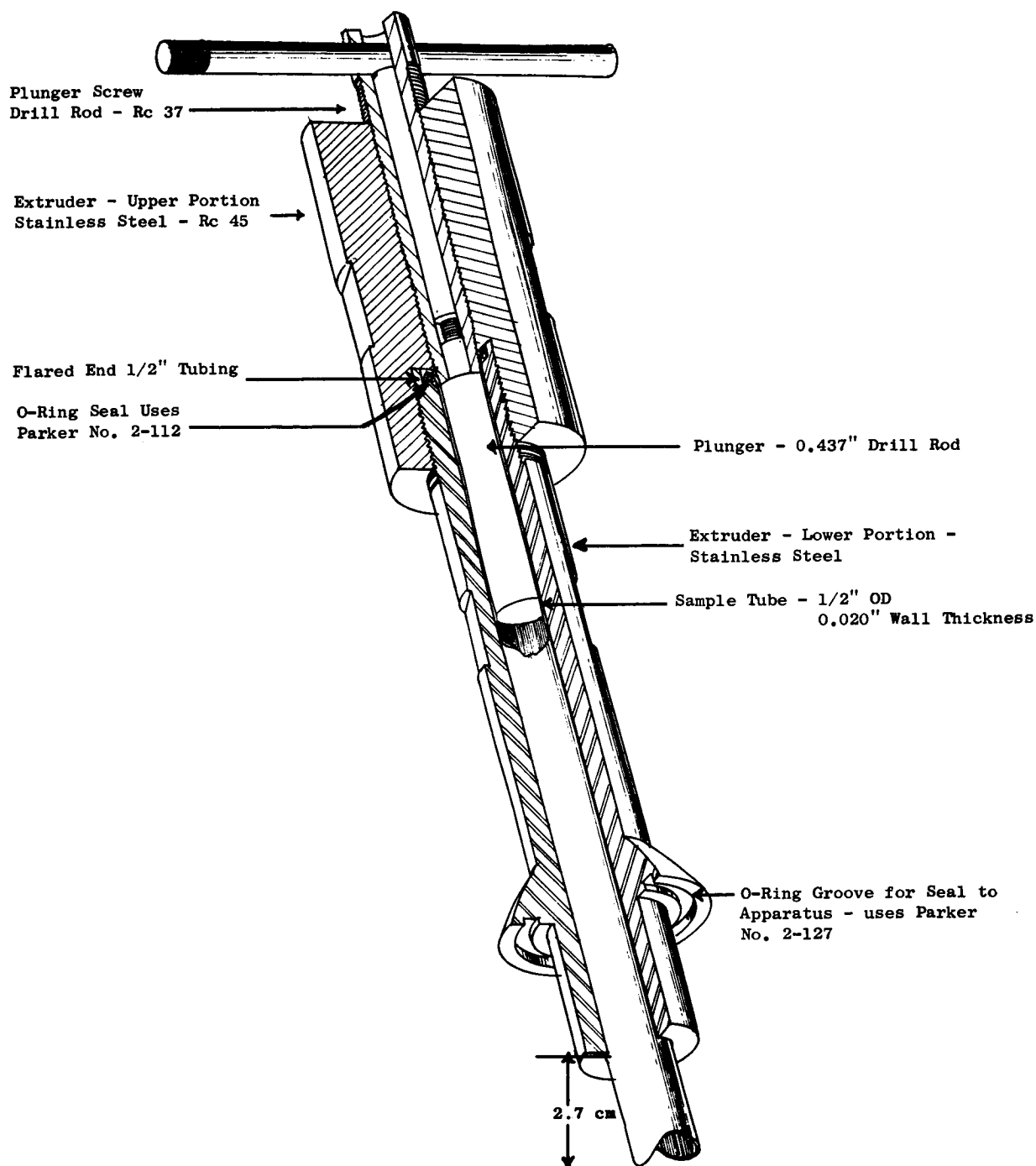


Figure 3.6.1.3. Alkali Metal Sample Extruder.

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Viewport, High Vacuum
OFHC Copper Gasketed

Viton and Bellows Sealed
VEECO Vacuum Valve

Sample Tube
Flush Reservoir

Bellows

3/8" Conoseal
Tube Union,
Female

Drain Valve,
Hoke TY445

To Vac-Inert Gas Manifold

Sample Tube

Modified Hoke HY441 Valve
or TY445 Valve

1/2" Conoseal Tube Union, Female,
To Fit Sample Source Or
Amalgamation Apparatus

Notes:

1. All parts austenitic stainless except viewport, Viton and copper gasket.
2. All SS connections TIG welded.
3. Drawing not to scale.

Figure 3.6.1.4. Alkali Metal Sampling Reservoir.

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3.6.1.1.4.1.1. Extruder Sample Preparation. One end of the sample tube is cut off with a tubing cutter as close to the Swagelok fitting as possible. This end of the tube is inserted into the bottom of the lower portion of the extruder and pushed up through the top. The O-ring (see Figure 3.6.1.3.) is placed over the tube, a small amount of alkali metal is removed, and the tube end is flared. The upper portion of the extruder is then screwed into place, forcing the flared tubing to seal against the top of the O-ring. Plunger(s) and plunger screw are then loaded and lowered until the potassium is contacted. The tube end protruding from the bottom is then cut off 2.7 cm below bottom of the lower position of the extruder. The extruder assembly is then attached to the reaction section of the analytical apparatus with a clamped O-ring joint.

3.6.1.1.4.1.2. Cutter Preparation. As shown in Figure 3.6.1.2., the cutter assembly is attached to the large glass O-ring fitting of the reaction flask. The cutter moves via a bellows for cutting samples and is positioned to place the cutter wire over the waste boat. The cutter wire is cleaned with water, hydrochloric acid, water, acetone, and air-dried, in this order. The cutter wire is heated by infrared lamps immediately prior to assembly.

3.6.1.1.4.1.3. Flask Preparation. The reaction flask is flushed thoroughly with hexane to remove all traces of mercury and grease. The flask is then washed in a "Sparkleen" solution, rinsed in water, then acetone, and then oven-dried. The stopcock is turned in with Apiezon "N" grease.

3.6.1.1.4.1.4. Stirring Bar Preparation. The nickel stirring bar is washed with nitric acid to remove mercury, rinsed with water, washed with hydrochloric acid, water, and acetone, and then dried in an oven.

3.6.1.1.4.1.5. Waste Boat Preparation. The titanium boat is cleaned of potassium waste metal, rinsed in hydrochloric acid, water, and acetone, and then oven dried.

3.6.1.1.5. Apparatus Assembly

3.6.1.1.5.1. Assembly. Referring to Figures 3.6.1.1. and 3.6.1.2., the apparatus is assembled as follows:

- a. Attach the cold trap to the vacuum-argon manifold with inert gas flowing.
- b. Insert the nickel stirring bar and titanium waste boat in the reaction flask and connect the reaction flask to the cold trap. The reaction flask must also be supported, as shown in Figure 3.6.1.1.
- c. Install the extruder, containing the sample tube, on the reaction flask.
- d. Attach the mercury reservoir to the reaction flask as shown in Figure 3.6.1.2. Support the mercury reservoir so that undue stress is not placed on the glassware.
- e. Connect the cutter system to the reaction flask and check to see that the cutter can be positioned over the titanium waste boat.
- f. Attach the amalgam receiver flask to the bottom of the reaction flask and support it with a lab jack.
- g. Turn off the inert gas flow and evacuate the apparatus to begin outgassing. If leaks are suspected, find them with acetone and eliminate them.

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h. Attach heating tapes and/or heating jackets to the apparatus in preparation for outgassing.

3.6.1.1.5.2. Outgassing. Referring to Figure 3.6.1.2., the glassware is heated by flaming with a gas burner to de-gas the interior surfaces of the flask and cold trap. The bellows and flange on the cutter assembly are heated by heat lamps. Due to the presence of solid alkali metal in the extruder, the outgassing temperature is limited to a temperature below the melting point of the metal to avoid melting it out of the sample tube.

3.6.1.1.5.2.1. Inert Gas Operation. For conduction of the analysis under inert gas, the system is outgassed until the pressure at the ion gauge shown in Figure 3.6.1.2. is 10^{-5} torr maximum while the system is cold. The pressure of $2-5 \times 10^{-6}$ torr is usually achieved within two hours.

3.6.1.1.5.2.2. Vacuum Operation. For analyses under vacuum, the system is outgassed until the pressure is 2×10^{-7} torr maximum and the pressure rise rate is less than 0.5 micron-liter per hour. This requires 24 to 48 hours of outgassing.

3.6.1.1.6. Analytical Procedure

3.6.1.1.6.1. Inert Gas Operation. When conducting the amalgamation analysis under inert gas, the cold trap between the analytical apparatus and the vacuum-inert gas manifold is put into use while the apparatus is cooling down after bakeout. (See Figure 3.6.1.1.). If argon is to be used, the cold trap is cooled by a dry-ice-acetone mixture or with liquid nitrogen if helium is to be used as the cover gas. With the cold trap in operation and the ion gauge indicating less than 5×10^{-6} torr pressure, the analytical apparatus is backfilled with inert gas to a pressure which exceeds atmospheric pressure by about 1/2 psi or one inch of mercury.

3.6.1.1.6.1.1. Extrusion Sampling - Sodium and Potassium. The solid alkali metal sample is obtained from the extruder as follows:

- a. The cutter wire is positioned to the left of the sample tube.
- b. About 1/2 inch of metal is extruded from the tube and cut off even with the end of the tube.
- c. This waste piece is then positioned over and melted off into the waste boat by infrared heat lamps or resistance heating of the cutter wire.
- d. The cutter is repositioned to the left of the sample tube, another 1/2-inch of metal is extruded, cut and melted into the waste boat.
- e. The cutter is repositioned to the left of the sample tube and the sample for analysis is extruded. The sample size is normally varied between 1 and 5 gms. or 3/4 to 3 3/4 inches long. The longer samples must be cut off in small pieces which are dropped individually into the reaction flask.
- f. The sample is cut, positioned over the center of the reaction flask, and melted off the wire into the bottom of the flask.

3.6.1.1.6.1.2. Liquid Sampling - Sodium, Potassium and NaK. For obtaining liquid samples, the reservoir shown in Figure 3.6.1.4. or a NaK container is connected to the analytical apparatus instead of the extruder.

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3.6.1.1.6.1.2.1. Liquid Sampling Apparatus. Figure 3.6.1.5. shows the apparatus used in obtaining a liquid sample. The liquid container is attached to the transfer line by a Conoseal, Swagelok or equivalent connector. The waste boat is positioned over the reaction flask and the cutter is placed to the right of the waste boat.

3.6.1.1.6.1.2.2. Liquid Sampling Procedure. For sodium and potassium, the sampling reservoir must be heated to melt the metal. Sodium should be heated to 300°F plus or minus 10°F, and potassium should be heated to 200°F plus or minus 10°F. A one-hour residence time at the recommended temperature has proven adequate to equilibrate the system and provide a homogeneous liquid sample. The sampling procedure is as follows:

- a. Open the valve on the reservoir (see Figure 3.6.1.4.) or the NaK container and flush the transfer line with 1 to 5 gms of liquid metal into the waste boat.
- b. Push the waste boat to the left with the cutter rod until the transfer line outlet can see the bottom of the reaction flask.
- c. Secure the cutter rod so that liquid does not touch it while taking the sample.
- d. Add the sample to the reaction flask dropwise, again using 1 to 5 gms of metal, using the same valve used to control the flushing of the transfer line.
- e. Allow sodium or potassium samples to cool and solidify before proceeding with the analysis.

3.6.1.1.6.1.3. Sample Amalgamation. The sodium, potassium or NaK sample should be amalgamated with the mercury as follows:

- a. Introduce mercury into the reaction flask slowly (to avoid smoking) until 40 ml of mercury have been added.
- b. With solid potassium samples, heat the mercury gently until the metal begins to melt on top of the mercury, and then react the metal with the mercury by gentle manipulation of the stirring bar. AVOID A VIOLENT REACTION of potassium with mercury by exercising care in heating the mercury.
- c. With sodium or NaK samples the amalgamation reaction is instantaneous and quite vigorous. For these metals it is not possible to float the material on top of the mercury. Therefore, the mercury is added in small increments until no further smoking occurs and then heated gently without agitation until the reaction is complete, as evidenced by no detection of solid metal with the stirring bar.

3.6.1.1.6.1.4. Extraction of the Alkali Metal from the Mercury

- a. Drain the initial alkali metal-rich amalgam from the reaction flask into 1N HCl until 1 to 2 ml of amalgam remain in the reaction flask. The quantity of HCl needed will depend on the sample weight and should be in excess of that needed to convert all the free alkali metal to the chloride.
- b. Add 20 ml of mercury to the reaction flask, heat gently to about 150°F and clean all metal droplets or particles from the flask walls with the nickel stirring bar.
- c. Again drain the amalgam into the 1N HCl used in (a) above, leaving 1 to 2 ml.
- d. Repeat steps (b) and (c) three more times; however, on the last extraction, the amalgam (mercury) is drained into 1 to 2 ml of distilled water containing 1 to 2 drops of phenolphthalein. If no red color results, it is assumed that all free alkali metal has been extracted from the reaction flask.

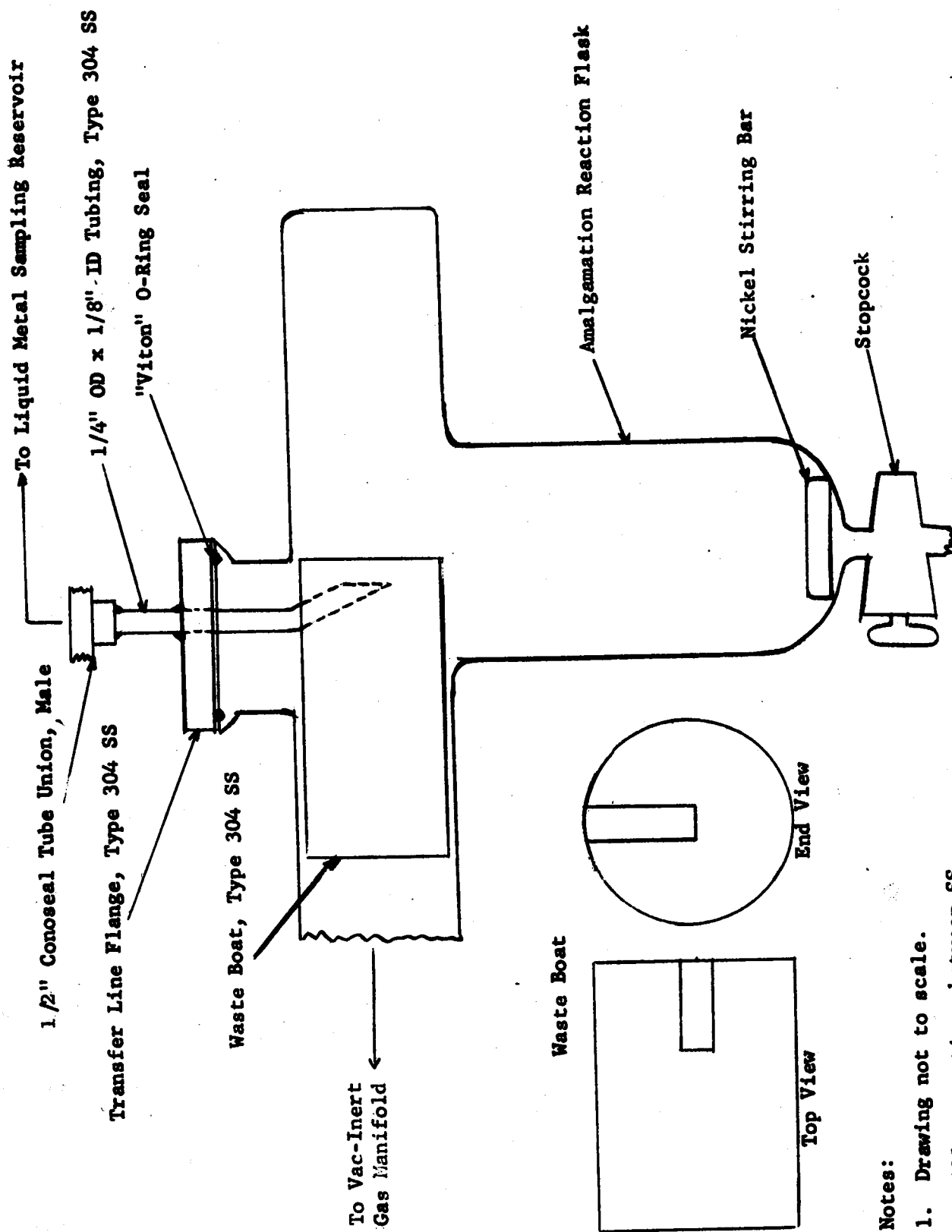
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Notes:

1. Drawing not to scale.
2. All connections between SS parts are TIG welded.

Figure 3.6.1.5. Basic Elements of Apparatus for Liquid Alkali Metal Sampling for the Amalgamation Method.

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3.6.1.1.6.1.5. "Oxide" Residue Removal. The "oxide" residue is then extracted from the reaction flask with distilled water of known pH and known volume. The water is sprayed over the interior walls of the flask and the stirring bar is used also to scrub the walls with water.

3.6.1.1.6.1.6. "Oxide" Residue Analysis. The residue solution is analyzed for oxygen content by both titrimetric and flame photometric methods.

3.6.1.1.6.1.6.1. Titrimetric Analyses

3.6.1.1.6.1.6.1.1. Titration Apparatus. Most analyses of alkali metals for oxygen by the amalgamation method are done on reasonably pure or purified metal. Thus the oxygen quantities normally found are small and require micro-techniques to obtain accurate titration data. The titration equipment used is described as follows:

The acid is added to the solution being titrated via a micro-buret syringe made by the Micro-Metric Instrument Co. The syringe has a volume displacement of 0.5 micro-liter of solution per each 0.001 inch of plunger travel and a total capacity of 0.5 ml of acid.

The syringe is operated by a Bird Kymograph which has speeds of 1, 0.2, 0.004, 0.008 and 0.0016 inch per minute.

The pH of the solution is measured by a Leeds and Northrup pH indicator, catalog No. 7664, which is connected in series to a Brown recorder where a trace of pH vs. time is recorded during the titration.

The solution being titrated is agitated with a Teflon covered stirring bar rotated by a magnetic stirrer.

3.6.1.1.6.1.6.1.2. Titration Procedure. The titration procedure is as follows:

a. The pH of the water used to extract the residue is first measured and the recorder is standardized.

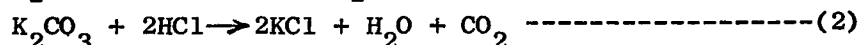
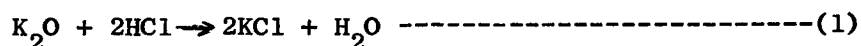
b. The micro-buret syringe is filled with 0.0125 N HCl normally, although stronger acid may be used when the oxide content of the residue is high.

c. The acid transfer tube and the pH electrodes are washed thoroughly with distilled water, then immersed in the residue solution, and the stirrer is turned on.

d. The recorder and Kymograph are actuated simultaneously and acid is added to the solution until the pH decreases to below the pH of the extraction water.

e. The pH vs. time trace is removed from the recorder and the oxygen content of the residue solution is calculated.

3.6.1.1.6.1.6.1.3. Titration Calculation. The oxygen content of the residue solution is calculated as follows: The determination of the oxygen content is based on the following two equations, using potassium monoxide as the example:



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From equation (1), one atomic weight of oxygen will react with two moles of HCl. From equation (2), one atomic weight of oxygen as K_2O from K_2CO_3 will react with two moles of HCl. Thus, although the carbonate may be present, the calculation of the oxygen is based on K_2O plus K_2O from K_2CO_3 .

Therefore, the oxygen amount is calculated as follows:

$$\left(\frac{8 \text{ gms O}}{\text{moles HCl}} \right) \times \text{liters HCl} \times \frac{\text{moles HCl(N)}}{\text{liter}} = \text{gms O} \text{ -----(3)}$$

where N = normality (usually 0.0125N or 0.125N HCl)

To determine the volume of HCl, the following formula is employed:

$$\left(\right) \text{ liters HCl} = \frac{C \times I \times T \times S}{1000} \text{ -----(4)}$$

where: C = recorder chart speed, minutes/inch
 I = distance between points on chart where pH is that of residue extraction water, inches
 T = microburet plunger speed, inches/minute
 S = microburet volume displacement, milliliters/inch

Inserting equation (4) into equation (3) gives:

$$\left(\right) \text{ gms O} = 0.008 \times C \times I \times T \times S \times N \text{ -----(5)}$$

Therefore:

$$\left(\right) \text{ ppm O} = \frac{0.008 \times C \times I \times T \times S \times N \times 10^6}{\text{sample weight, gms}} \text{ -----(6)}$$

3.6.1.1.6.1.6.2. Flame Photometric Analysis. An alternate method of measuring the amalgamation residue makes use of flame emission photometry. Any commercial flame photometer capable of isolating and measuring the appropriate line emission of the alkali metal of interest may be used. The Beckman DU Spectrophotometer has been found to be quite satisfactory for this purpose. The general flame analytical procedure is as follows:

- a. Calibration curves are prepared over various concentration ranges of NaCl and KCl in the distilled water used for residue extraction. The spectrographically pure NaCl and KCl used in making the known standard solutions are obtained from Johnson, Matthey & Co., Limited in London, England.
- b. The total potassium and sodium contents of a given residue extraction solution are then determined by transmission measurements, and the calibration curves are based on known standards.
- c. The assumption is then made that all potassium and sodium were originally present as monoxide and the oxygen contents are calculated on this basis.

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3.6.1.1.6.1.7. Sample Weight Determination. The sample weight is determined by back titration of the acid solution produced by the reaction of the alkali metal amalgam with the 1N HCl as described in 3.6.1.1.6.1.4. The end point indicator used is phenolphthalein. 1N NaOH is used for the back titration. The sample is determined from the following formula:

$$\text{Sample weight, grams} = (\text{ml HCl} - \text{ml NaOH}) \frac{(\text{mol. wt. of alkali metal})}{1000}$$

3.6.1.1.6.2. Vacuum Operation. For vacuum operation, both cold traps shown on the apparatus in Figure 3.6.1.1. are cooled with liquid nitrogen. Otherwise, the analytical technique is the same as for inert gas with a few minor differences. The vacuum amalgamation technique has been shown to be capable of producing results for oxygen in potassium which compare fairly well with results obtained on similar samples by the amalgamation procedure run under inert gas; however, the vacuum technique does present the following problems. Mercury is deposited in the upper, colder regions of the reaction flask, making post-analytical cleanup a greater problem. The amalgamation reaction also tends to be more violent under vacuum. Any alkali metal hydrides may be partially decomposed at the amalgamation temperature under vacuum.

3.6.1.2. Determination of Oxygen in Lithium

3.6.1.2.1. The Fast Neutron Activation Method. At present the fast neutron activation technique is the only approved method for determining oxygen in lithium metal, and the only approved analytical laboratory is Activation Analysis Service, General Atomic Division, General Dynamics, P. O. Box 608, San Diego, California 92112.

3.6.1.2.1.1. Specimen Preparation. Specimens of lithium metal should be obtained as described in Sections 3.7.3.1., 3.7.4.1.1. and 3.7.4.2. of this specification. The stainless steel sample tube should be capped tightly with stainless steel Swagelok (or equivalent) caps prior to shipment.

3.6.1.3. Determination of Oxygen in Rubidium. Same as Section 3.6.1.2.

3.6.1.4. Determination of Oxygen in Cesium. Same as Section 3.6.1.2.

3.6.2. Preparation of Samples for Determination of Metallic Impurities in Alkali Metals.

3.6.2.1. Sodium, Potassium and NaK. Spectrographic analysis of potassium, sodium, or NaK for metallic impurities is done on the chloride salts of the alkali metals. The chloride salts are prepared as follows:

a. A platinum dish is cleaned by melting potassium pyrosulfate in the dish with agitation to assure that the entire platinum surface is contacted by the molten salt. The residue is then washed from the dish with distilled water and oven-dried at 100-120°C.

b. The alkali metal sample (1-2 gms) is then introduced into the clean platinum dish. The metal may be extruded, cut and placed in the dish as a solid, or it may be introduced in liquid form. In any case, the method of placing the metal specimen into the dish should be such that no metallic contaminants are added to the sample.

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c. Cover the alkali metal sample with spectroquality hexane (DO NOT USE PRACTICAL GRADE HEXANE), and add absolute ethanol to the hexane until the alkali metal is converted to the ethoxide. FACE SHIELD AND GLOVES SHOULD BE USED DURING DISSOLUTION OF THE METAL FOR PERSONNEL PROTECTION.

d. Add concentrated reagent grade HCl to the ethoxide-ethanol-hexane solution dropwise to convert all ethoxide to the chloride. If HCl is ADDED TOO RAPIDLY, THE HEAT OF THE CONVERSION REACTION WILL IGNITE THE FLAMMABLE SOLUTION.

e. Place the chloride solution on a hot plate or steam bath and evaporate to dryness in a fume hood.

f. Store the resulting alkali metal chloride in a clean glass vial until the spectrographic analysis is done.

3.6.2.2. Lithium. Spectrographic analysis of lithium for metallic impurities is performed on the carbonate, Li_2CO_3 . The carbonate is prepared as follows:

a. Same as Section 3.6.2.1. a

b. Same as Section 3.6.2.1. b

c. Same as Section 3.6.2.1. c

d. Add distilled water to the ethoxide-ethanol-hexane solution dropwise to hydrolyze the ethoxide. Then bubble CO_2 through the solution using a non-contaminating tube; i.e., platinum; or cover with CO_2 until the pH of the aqueous solution is reduced to 8 or 9. Dry ice is a convenient source of CO_2 and is sometimes used to convert LiOH to Li_2CO_3 .

e. Evaporate to dryness.

f. Store in a clean, dry, glass vial

3.6.3. Spectrographic Methods for Determining Metallic Impurities in Alkali Metals

3.6.3.1. Potassium

3.6.3.1.1. Specimen Preparation. Metallic impurities in potassium metal are usually determined on the chloride salt of potassium and the impurity levels are reported as ppm impurity in potassium chloride. The procedure for converting potassium metal to potassium chloride is described in paragraph 3.6.2.1. of this specification.

3.6.3.1.2. Analytical Procedure. This method provides for the quantitative determination of 20 impurity elements in the range 25-250 ppm in potassium chloride prepared from potassium metal. The method can also be used for obtaining a semi-quantitative estimate of the 20 impurity elements in the range 1-25 ppm by varying conditions as outlined below.

3.6.3.1.2.1. Apparatus and Reagents

a. Jarrel-Ash 3.4-meter Wadsworth Mounting Spectrograph.

b. Excitation Source, NSL Spec - Power.

c. Neutral Density Filter, Steps % Transmission, 100-64.3-42.5-27.0-17.5-11.3-7.1.

d. Comparator microphotometer, Jarrell-Ash Model SA-200.

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- e. Photographic plate processing unit, NSL Spec. Processor.
- f. Spectrographically pure KCl.
- g. Spectrographic Standards, Jarrell-Ash Master SQ powder standards.
- h. Graphite Electrodes.

3.6.3.1.2.2. Preparation of Standard Curves. Spectrographic Standards for establishing analytical curves are prepared by mixing Jarrell-Ash "Master SQ Standards" with spectrographically pure KCl. Fifteen milligrams of standards containing from 25 to 250 ppm of the impurities of interest are weighed directly into graphite cup electrodes. The loaded electrodes are dried at 115°C for 30 minutes. The dried, loaded electrodes are stored in a dessicator until arced. Details of the analytical apparatus and operating parameters are summarized in Table 3.6.3.1. Using the established conditions, the samples are arced for the designated times and the spectra recorded on photographic plates.

TABLE 3.6.3.1.

APPARATUS AND CONDITIONS

Spectrograph	Jarrell-Ash 3.4 m Wadsworth
Wavelength Range, A	2400-4250
Slit Width, μ	30
Slit Height, mm	2.8
Filter	Neutral density, steps %T, 100-64.3-42.5-27.0-17.5-11.3-7.1
Excitation Source	NSL Spec-Power
DC Amps	8.0
Electrodes	
Sample	UCP-105-S
Counter	UCP-2615
Analytical Gap, mm	4
Gas Mixture to Stallwood Jet	50-50 Argon-Oxygen, Burdett Oxygen Co.
Flowrate, SCFH	8
Exposure	
Pre-Burn	None
Exposure	Total Burn
Emulsion	Kodak SA-1, developed in D-19 for 4 minutes at 68°F

Intensity ratios based on the 3446.722A line of potassium are determined for the standards using the comparator-microphotometer and a Seidel calibration curve. The analytical line pairs for each of the elements of interest are given in Table 3.6.3.2. The final analytical curves are obtained by plotting the intensity ratios against impurity concentration on semi-log scales.

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TABLE 3.6.3.2.

ANALYTICAL LINE PAIRS
FOR THE IMPURITY ELEMENTS SOUGHT

<u>Element</u>	<u>Element Line, A^a</u>	<u>Element</u>	<u>Element Line, A^a</u>
Boron	2497.733	Sodium	3302.323
Manganese	2801.064	Silver	3382.891
Magnesium	2852.129	Zirconium	3391.975
Lead	2833.069	Cobalt	3405.120
Silicon	2881.578	Nickel	3414.765
Iron	3020.640	Chromium	3605.333
Vanadium	3102.299	Titanium	3635.463
Molybdenum	3132.594	Aluminum	3961.527
Tin	3175.019	Calcium	3968.468
Copper	3247.540	Columbium	4058.938

^aPotassium internal standard line 3446.722 A with filter, 11.6%T.

Impurity levels below 25 ppm yield lines of very low intensity and it is not practical to establish analytical curves for impurities below 25 ppm.

3.6.3.1.2.3. Analysis of Samples. Fifteen milligram samples of potassium chloride which have been converted from potassium metal according to the procedure detailed in 3.6.2.1. of this specification are loaded into graphite cup electrodes. The loaded electrodes are dried for 30 minutes at 115°C and stored in a dessicator until arced. The conditions for burning the unknown samples are identical to the conditions used to establish the analytical curves. Three exposures are required for each sample.

3.6.3.1.2.4. Calculations

- a. Determine the average per cent transmission of the three exposures for each analytical line of interest.
- b. Determine the concentration of each element based on KCl from the simultaneously prepared standard curve.

NOTE: For impurity levels from 1-25 ppm a semi-quantitative estimate of impurity concentration is possible by visually comparing line intensity of low level standards. This method should have an accuracy of ± 5 ppm. Table 3.6.3.3. shows the detection limits and analytical line pairs for low level impurities.

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TABLE 3.6.3.3.

SENSITIVITY OF DETECTION OF ELEMENTS IN POTASSIUM (AS CHLORIDE)

Element	Line Observed, A	Minimum Detected Amount
Mg -----	2802.70 -----	<1
Mn -----	2794.82 -----	<1
Cr -----	4254.35 -----	<1
Sn -----	2863.33 -----	1
Si -----	2516.12 -----	<1
Ti -----	3653.50 -----	1
Al -----	3082.16 -----	<1
V -----	3102.30 -----	5
Cu -----	3247.54 -----	<1
Ag -----	3280.68 -----	<1
Co -----	3405.12 -----	1
Ni -----	3414.77 -----	1
Zr -----	3601.19 -----	5
Pb -----	3639.58 -----	5
Mo -----	3902.96 -----	<1
Ca -----	3933.67 -----	<1
Cb -----	4079.73 -----	1
B -----	2497.73 -----	5
Fe -----	3719.94 -----	1
Na -----	3302.99 -----	1
Ba -----	Not detected up to 25 ppm	

3.6.3.1.2.5. Precision and Accuracy. In order to evaluate the accuracy and precision associated with the method, a 100 ppm standard was prepared by mixing a Jarrel-Ash Master SQ standard with pure KCl. This standard was arced 4 times under the prescribed conditions and per cent standard deviation was calculated for each element of interest. These data are summarized in Table 3.6.3.4.

3.6.3.2. Sodium

3.6.3.2.1. Specimen Preparation. See paragraph 3.6.2.1. of this specification.

3.6.3.2.2. Analytical Procedure. This method is under development, but will be essentially the same as for potassium (See paragraph 3.6.3.1.2.). Until such time that the method is established, samples may be analyzed by Nuclear Materials Equipment Corporation, Apollo, Pa.

3.6.3.3. Lithium

3.6.3.3.1. Specimen Preparation. Metallic impurities are determined in lithium carbonate prepared from the lithium metal, and the impurity levels are reported as ppm in Li_2CO_3 . The technique used to prepare the lithium carbonate is described in paragraph 3.6.2.2.

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TABLE 3.6.3.4.PRECISION AT 100 PPM LEVEL

<u>Element</u>	<u>Per Cent Standard Deviation*</u>	<u>Element</u>	<u>Per Cent Standard Deviation</u>
Boron	± 49.9	Sodium	± 32.6
Magnesium	± 7.7	Silver	± 17.1
Manganese	± 7.3	Cobalt	± 7.5
Lead	± 9.3	Nickel	± 13.4
Silicon	± 25.0	Chromium	± 13.2
Iron	± 15.9	Zirconium	± 11.6
Vanadium	± 25.5	Titanium	± 6.2
Molybdenum	± 12.2	Aluminum	± 38.4
Tin	± 15.5	Columbium	± 8.4
Copper	± 22.1	Calcium	± 50.0

* Per cent Standard Deviation in this method is calculated as follows:

$$\% \text{ Std. Dev.} = \frac{100}{C} \times \sqrt{\frac{\sum d^2}{n - 1}}$$

Where: C = Average concentration in ppm

d = Difference of the determination from the mean

n = Number of determinations

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3.6.3.3.2. Analytical Procedure. This method is under development, but will be essentially the same as for potassium (see paragraph 3.6.3.1.2.). Until such time that the method is established, samples may be analyzed by Nuclear Materials Equipment Corporation, Apollo, Pa.

3.6.4. Analytical Methods for the Determination of Nitrogen in Alkali Metals. These methods are under development. Until such time that methods have been established samples may be analyzed by Ledoux and Company, Teaneck, New Jersey or by Mine Safety Appliance Research, Callery, Pennsylvania.

3.6.5. Analytical Methods for the Determination of Carbon in Alkali Metals. These methods are under development. Until such time that methods have been established samples may be analyzed by Ledoux and Company, Teaneck, New Jersey or by Mine Safety Appliance Research, Callery, Pennsylvania.

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3.7. Alkali Metal Sampling

3.7.1. Sampling Objectives. The general objective sought in an alkali metal sample is the homogeneous, representative, uncontaminated specimen which can be analyzed as accurately as possible for the desired impurity(s) concentration. Specifically, the objectives of this sampling specification are:

a. To indicate the sampling criteria for obtaining alkali metal specimens to be analyzed for oxygen, carbon, nitrogen and metallic impurities.

b. To specify types of apparatus which can be used for taking samples

c. To present semi-detailed procedures for sample taking

3.7.2. Sampling Criteria3.7.2.1. For Oxygen Analysis

a. When sampling high purity metal under inert gas, the purity of the gas must be such that the contamination of the sample by the gas does not exceed 1 ppm oxygen as the monoxide.

b. When sampling high purity metal under vacuum, the pressure rise rate of the sampling system must be such that the contamination of the sample therefrom does not exceed 1 ppm oxygen as monoxide.

c. When sampling liquid sodium or potassium with a suspected oxygen concentration higher than the solubility for oxygen of the metal at its melting point, it is recommended that the specimen be obtained horizontally if the analytical specimen is to be sampled later as a solid. Regardless of the sampling position used for this grade of metal, the entire sample should be analyzed and the results averaged to off-set possible uneven distribution of oxygen in the sample. Sampling temperature should be high enough to dissolve all oxide present in the metal source.

d. The transfer lines between the alkali metal source and the sampling system should be flushed with a quantity of metal twice the volume of the transfer line before sampling. The sample tube should be flushed with a quantity of metal twice the volume of the tube after the transfer line has been flushed.

e. All samples should be transferred and taken in austenitic stainless steel or other material which does not interfere with the analytical procedure and results.

f. Where high accuracy is not required or under other conditions where all the previously stated criteria cannot be met, the criteria may be waived with the approval of the person responsible for control and measurement of alkali metal purity (the Alkali Metal Custodian).

3.7.2.2. For Carbon Analysis. The essence of the criteria indicated for oxygen samples applies to samples to be analyzed for carbon except that such species as CO or CO₂ or other compounds which would contaminate the sample should not be present in the sampling system to the extent that 1 ppm carbon contamination of the sample would occur.

3.7.2.3. For Nitrogen Analysis. The essence of the criteria presented in paragraph 3.7.2.1. applies to samples for nitrogen analysis except that nitrogen or its compounds should be excluded from contact with the sample so that contamination by nitrogen does not exceed 1 ppm nitrogen.

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3.7.2.4. For Metallic Analyses. The criteria by which samples should be obtained for metallic analyses by spectrographic techniques include:

a. Samples with oxygen content greater than 100 ppm should not be taken in austenitic stainless steel at temperatures in excess of 500° F if true concentrations of the elements in the sample tubing are desired, since corrosion of the austenitic stainless steel tubing could occur at higher temperatures.

b. Conversion of sodium, potassium or NaK to their chlorides should be done in a freshly pyrolyzed (potassium pyrosulfate) platinum dish with spectroquality hexane, absolute ethnaol, and reagent grade concentrated HCl. See paragraph 3.6.2.1.

3.7.3. Sampling Apparatus. The apparatus used for sampling sodium, potassium and NaK for oxygen, carbon and nitrogen analysis can be the same for all three metals except that NaK must be contained between valves since it is liquid at room temperature. The sodium and potassium may be introduced into the analytical apparatus in either solid or liquid form -- the NaK in liquid form only. The solid samples are extruded the liquid samples are taken from a leak-tight container. Thus, two types of sampling apparatus are required depending on whether solid or liquid samples are to be analyzed.

3.7.3.1. Apparatus for Solid Samples. Solid samples are obtained by extruding the metal from austenitic stainless steel tubing or by cutting metal containing sections from the tubing. For extrusion, the sample tube must be 1/2-inch outside diameter by 20-mil wall thickness and at least 10 inches long. Where sections of tubing are cut to obtain the metal sample, the tubing wall thickness is not important, but the other dimensions should approximate those used for extrusion samples.

The apparatus system used for obtaining solid samples can be either of two types. The two types are shown schematically in Figure 3.7.3.1. The apparatus for sampling large alkali metal sources utilizes a flush container large enough to clear the transfer line, and the sampling system is positioned between the source and flush container as shown.

The apparatus used for small sources utilizes the sampler overflow volume as the flush container.

For both systems shown in Figure 3.7.3.1., all valves from which alkali metal must be cleaned are Hoke HY473A angle pattern, and the connecting fittings are either Swagelok, Gyrolok, Conoseal or equivalent type connectors. As shown, both systems are equipped with argon or helium and/or vacuum manifolds for obtaining a pure inert gas atmosphere or high vacuum for sampling.

3.7.3.2. Apparatus for Liquid Samples. The sampler from which liquid samples will be introduced into analytical apparatus will be placed in the same location shown in Figure 3.7.3.1. for solid samples. However, the liquid sampling containers must be completely isolated by valves after the sample is taken, and the valve which must be attached to the analytical apparatus must be cleanable up to the seat. Figure 3.6.1.5. shows the type of apparatus from which liquid samples will be taken.

It should be noted that the sampling criteria stated in paragraph 3.7.2. may be more difficult to meet when liquid samples are used, since the molten metal will more actively getter and dissolve contaminants present in the sample container. Therefore, extreme attention to cleanliness, of cover gas and sample tube, as well as to pressure rise rate under vacuum conditions, is necessary if the sample is to be introduced into the analytical apparatus in liquid form.

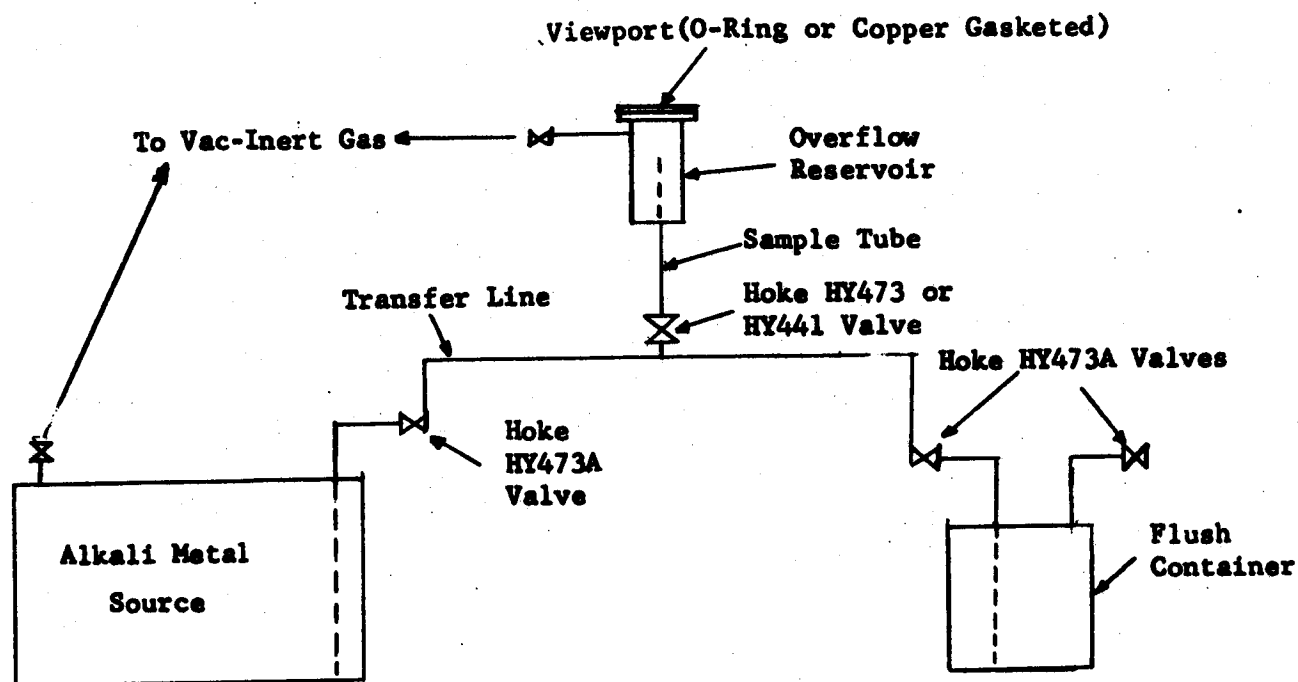
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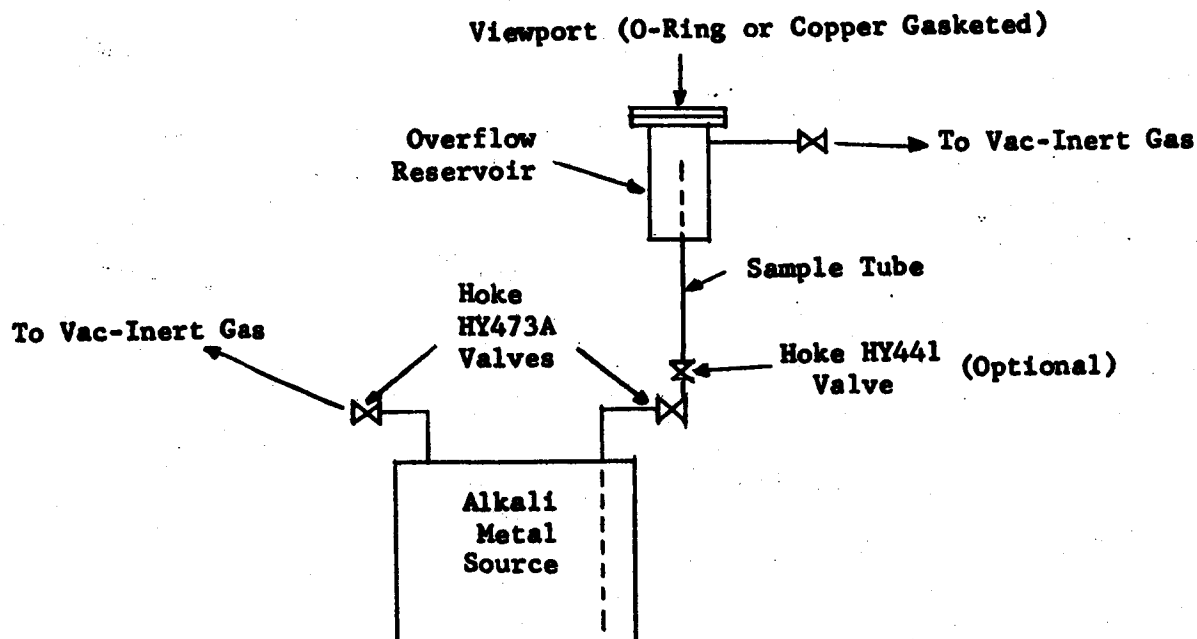
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Sampling Rig Where Large Flush Quantities are Required



Sampling Rig Where Small Flush Quantities are Required

Figure 3.7.3.1. Alkali Metal Sampling Arrangements.

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3.7.4. Sampling Procedures. The procedures used in taking samples for analysis will include preparation of apparatus and the operations required for obtaining representative samples for quantitative analyses.

3.7.4.1. Sample Apparatus Preparation

3.7.4.1.1. For Extrusion Sampling (Na and K). Referring to Figure 3.7.3.1. the following operations are required to prepare the sampling apparatus:

- a. Degrease and pickle the sample tube, overflow reservoir and connecting fittings with 20% HCl for 10 to 15 minutes, rinse with water, acetone and dry.
- b. Attach the sample tube and viewport cover to the overflow reservoir.
- c. Attach the sampling assembly to the clean Hoke HY437A sampling valve or tee in the flush line and connect the inert gas-vacuum line to the gas valve on the reservoir.
- d. Evacuate the sampling system to less than 10^{-3} torr and leak-check cold to meet the criteria stated in paragraph 3.7.2.
- e. Heat the sampling system to 250-300°F (or higher, if necessary to obtain a representative sample) and outgas at less than 10^{-3} torr until the pressure rise rate meets the contamination criteria stated in paragraph 3.7.2.

3.7.4.1.2. For Liquid Sampling. Referring to Figure 3.6.1.5., the initial preparation (pickling) of the liquid sampling apparatus will be done as in paragraph 3.7.4.1.1., except that this system is designed for high vacuum, high temperature use. The sample tube proper is welded to the overflow reservoir and to a Hoke HW441 valve on the bottom. Thus, pickling and cleanup of the interior surfaces of the system will be done during fabrication.

Following assembly of this system, a helium leak-check is required with a maximum leakage of 5×10^{-10} std. cc/sec of air allowed.

Attach the sampler to the alkali metal source and outgas at less than 10^{-5} torr at the temperature at which the sample will be taken until the pressure rise rate meets the previously specified requirements.

3.7.4.2. Sampling Operations. With sampler attached, the sampling procedure will be as follows:

- a. Flush the transfer line and sample tube with the required volume of metal at the required temperature. This operation may be done under high vacuum or pure inert gas for either extrusion or liquid samplers.
- b. For extrusion samples, pressurize the sampler to 5 psig, cool to room temperature, remove the sampler and cap the end exposed to air.
- c. For liquid samples, close the valve on the bottom of the sampler, pressurize to 5 psig, cool to room temperature and remove the sampler from the source. Clean the valve on the bottom of the sample tube to the seat as specified in paragraph 3.10.1.

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3.8. Monitoring

3.8.1. Definition. Monitoring, as used in this specification, is the procedure(s) by which the purity of the alkali metal in use in a system is assured.

3.8.2. Purpose. The purpose of monitoring procedures is to maintain the impurity concentration levels in the alkali metals below certain specified limits.

3.8.3. Justification. Justification of the effort, time and money required by a monitoring program is based on the known or suspected deleterious effects which certain impurities may have on the structural integrity and/or operation of facilities or equipment using alkali metals as working fluids. Some specific effects due to contaminants are given below; however, insufficient data are available to ascertain whether or not certain impurities are harmful, and as a consequence, the overall policy is to use liquid metals of the highest practical purity in order to avoid the problem as far as is possible.

3.8.3.1. Deleterious Effects Due to Impurities

3.8.3.1.1. Oxygen. A number of effects have been noted which are due to oxygen contamination in the alkali metals.

3.8.3.1.1.1. Plugging. When the temperature in a portion of a system drops to a point where the solubility limit of a dissolved impurity is exceeded, precipitation will occur and may cause a plug. The oxide may be either the alkali metal oxide or a more complex oxide such as sodium columbate.

3.8.3.1.1.2. Corrosion. It is known that alkali metals containing oxygen react with containment materials such as stainless steel, nickel, columbium, etc. and with titanium and zirconium in a way which may lead to the dissolution or spalling of the metal surfaces. Such attack may weaken the containment material and/or yield particulate matter which may plug or abrade various components.

3.8.3.1.1.3. Mass Transfer. The presence of as little as 50 ppm oxygen in sodium has been associated with the mass transfer of carbon in austenitic and ferritic steel systems. It is also known that columbium may be transferred as a columbate when oxygen is present. Stainless steel loops circulating sodium at 650°C show dissolution losses to be ten times as great at 30 ppm oxygen in sodium as for 5 ppm oxygen in sodium.

3.8.3.1.1.4. Embrittlement. The presence of oxygen may lead to oxygen embrittlement in various alloys, especially refractory alloys.

3.8.3.1.2. Carbon. At least two effects have been noted which are associated with carbon in alkali metals.

3.8.3.1.2.1. Carburization. It has been noted that as little as 40 ppm carbon can lead to very rapid carburization of stainless steels at temperatures in the vicinity of 1200°F provided there is a source of carbon present. When no source is present, carburization ceases but the carbon content does not decrease drastically - perhaps to only 20 or 30 ppm. The mass transfer of stainless steel is also increased by the addition of carbon (10 to 15 ppm) to sodium.

3.8.3.1.2.2. Reduction of Getter Efficiency. The formation of carbide on the surfaces of titanium or zirconium getters in alkali metal systems has been found to reduce the efficiency of the getter for the removal of oxygen.

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3.8.3.1.3. Nitrogen. Nitrogen contamination in the alkali metals can also lead to reduction of getter efficiency for oxygen removal by the formation of nitrides on titanium and zirconium surfaces. Nitrogen can lead to embrittlement of various metals. Sodium and potassium do not normally dissolve nitrogen to any great extent; however, lithium does.

3.8.3.1.4. Hydrogen. The presence of hydrogen in alkali metals can lead to hydrogen embrittlement, especially of refractory metals. However, the test temperatures of systems constructed of these materials generally exceed the decomposition temperatures of the hydrides of most materials and the hydrogen diffuses out of the system, so it is generally not a problem.

3.8.3.1.5. Calcium. The presence of calcium in alkali metals has been associated with nitrogen transfer and has lead to a less than 50 ppm specification on Reactor Grade Sodium.

3.8.3.1.6. Metals. The presence of other metals as contaminants in the alkali metals is generally associated with the presence of oxygen. Since these oxides are not detected by the usual analytical methods for oxygen in the alkali metals and since they may cause oxygen transfer or plugging it is necessary to analyze the alkali metals for other metals.

3.8.3.2. Purification Efficiency. Monitoring the impurity levels before and after purification operations, such as cold trapping or hot trapping, will indicate how effective the purification step was, and thus provide the operator with information which is of value in determining whether the cold trap or hot trap is saturated, or whether the temperatures or flow rates through these devices should be altered.

3.8.4. Monitoring Procedures. The specific procedure by which the impurity levels of the alkali metal(s) in a particular system will be monitored is the responsibility of the Program Manager; however, it will be necessary to comply with the following essential requirements.

3.8.4.1. Lithium, Sodium, Potassium, and NaK

a. Analyze the as-received metal, before transfer to the system, for oxygen and metallic impurities, and carbon also when it is of particular concern. Lithium must also be analyzed for nitrogen in addition to oxygen and metallic impurities.

If it does not meet the quality specification, it must be purified or replaced.

b. If the alkali metal must be purified, it must be re-analyzed afterward.

c. When a system is capable of being flushed prior to operation it should be flushed and the alkali metal re-analyzed for oxygen and metallics; and repurified or replaced if necessary. This process of flushing, purification and analysis must be continued until the impurity levels of the alkali metal fall below the maximum limits specified. Lithium must also be analyzed for nitrogen.

d. Whenever, an accident occurs or components are changed in a way which could possibly lead to the exposure of the alkali metal to the atmosphere or other contaminants, the alkali metal must be re-analyzed and repurified or replaced if necessary.

e. In the case of systems where the exposure of the alkali metal to oil or other contaminants is possible, analyses should always be made for this contaminant also.

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f. The alkali metals used in working systems, such as heat transfer, turbine, and bearing and seal test rig loops should be analyzed periodically for oxygen and metallic impurities, and in the case of lithium for nitrogen. Such periodic analyses will generally be performed between testing periods. They will provide information on the condition of the loop such as excessive corrosion and leakage between primary and secondary circuits. Continuous oxygen monitoring devices such as plugging meters may be used when required for a particular facility.

3.8.4.2. Rubidium and Cesium. If rubidium and cesium are used in loop systems, the same monitoring procedures should be used as for the other alkali metals (see previous section). However, when they are used in such devices as ion engines, it is generally impractical to analyze the metal after its introduction to the feed system, but its purity should be established prior to the introduction. Historically, the vendor's analysis has been accepted and this practice will be continued except when there is some reason to suspect that the alkali metal has been contaminated. If it is discovered that contaminants which would be deleterious to the engine or the feed system are present, the metal should be purified or replaced.

3.8.4.3. Static Systems. Static systems include such devices as capsules, wick boilers, the alkali metal wetting study apparatus, etc. The purity of alkali metals used in such systems must be established before introduction to the device. When an inert gas chamber or vacuum chamber is used during the transfer of the metal, a sample should be obtained in the chamber during the transfer, under conditions which are as similar to the transfer conditions as is practical. The sample container should be sealed in the chamber and the sample analyzed to determine the quality of the metal placed in the device.

3.8.5. Maximum Allowable Impurity Concentrations. Several programs use alkali metals which are specially purified, for which the purity requirement is extremely high and for which special purification and purity specifications have been written. All other programs should maintain the purity of the alkali metals in use so that they comply with the following specifications.

3.8.5.1. Lithium. Specification 01-0030-00-A High Purity Lithium Metal, Table I.

3.8.5.2. Sodium. Specification 01-0032-00-B, Hot Trapped Reactor Grade Sodium Metal, except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.3. Potassium. Specification 01-0034-00-B, Hot Trapped High Purity Grade Potassium Metal, except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.3. Eutectic NaK. Specification 01-0050-00-A Hot Trapped High Purity Grade Eutectic NaK except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.4. Rubidium. Specification 01-0051-00-A High Purity Grade Rubidium Metal.

3.8.5.5. Cesium. Specification 01-0052-00-A, High Purity Grade Cesium Metal.

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3.9. Handling

3.9.1. Definition. Handling, as used in this specification, refers to the methods and techniques used to transfer alkali metals from one container, apparatus or device to another and to determine the quantity transferred.

3.9.2. Purpose. The purpose of this section is to describe methods by which alkali metals may be transferred from one container to another with a minimum of contamination, and the ways in which the quantity transferred may be determined. It is not the intention to limit the methods used solely to those described here, but instead to provide those involved with alkali metals with several procedures which can serve as guides.

3.9.3. Transfer Methods

3.9.3.1. Transfer in Air. It is sometimes desirable to transfer small quantities of solid alkali metal at low temperatures without resorting to complex handling systems. This can be done by using an inert cover liquid of high purity which does not dissolve appreciable quantities of atmospheric constituents such as nitrogen, oxygen, carbon dioxide and water vapor. Hydrocarbon liquids such as high purity mineral oil or spectrographic grade hexane are generally used for this purpose. When the proper care is exercised, it is possible to handle alkali metals for brief periods under such liquids without excessive contamination. Care should be taken that these flammable liquids do not catch fire. Also, alkali metals which may be coated with peroxides or superoxides must not be placed in such solvents since they may react violently with such active oxides.

This technique is not recommended for use with liquid alkali metals. Also, it should be remembered that lithium is less dense than the hydrocarbon liquids and so will float.

3.9.3.2. Transfer in Inert Gas and Vacuum Chambers

3.9.3.2.1. Inert Gas. Inert gas chambers or glove boxes are often used to facilitate the transfer of both liquid and solid alkali metals. If it is important not to contaminate the alkali metal, it is generally concluded that the inert gas should contain no more than 1 molar ppm of any particular active impurity, and that even then the exposure time should not exceed a few hours. It is not adequate to put high purity gas in the chamber and use it statically because outgassing of the chamber walls and diffusion of the atmosphere through the gloves contaminates the gas to unacceptable levels within a few minutes. A continuous purification system should be used through which the gas is recirculated at a rate which has been proved to be rapid enough to maintain the gas at the acceptable impurity level.

3.9.3.2.2. Vacuum. Alkali metals may be transferred in a vacuum chamber using remote handling techniques at pressures up to 10^{-5} torr provided the exposure time is not too long. Capsule filling operations which required less than two hours at about 10^{-5} torr have resulted in oxygen contamination to the extent of 5 to 10 ppm on occasion.

A pressure of 10^{-5} torr is equivalent to an inert gas containing about 10^{-2} molar ppm of impurities. The reason that one can use an inert gas of much greater impurity level is that the rate of contamination is slowed because it is necessary for the impurities to diffuse to the surface of the alkali metal.

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3.9.3.3. Transfer Using Inert Gas Pressure. This is the most used method for transferring liquid alkali metals. The technique is, in essence, that of pushing the liquid metal from one container to another through a connecting pipe. Usually the empty container is evacuated while the full container is pressurized to a few psig. Containers, valves and piping must be fabricated of corrosion resistant materials such as stainless steel, columbium, tantalum, etc. Valves should be of the bellows sealed, packless type; and if disconnects are to be made after the passage of alkali metal through them, they should have an angle pattern and should be oriented so that they may be cleaned back to the valve seat using the procedure described in paragraph 3.10.1 of this specification.

For critical applications where the highest purity is required, all parts of the system must be carefully cleaned by degreasing, pickling, rinsing with distilled water and drying under conditions which do not recontaminate the component prior to assembly. The components must be connected using approved welding techniques such as those described in Specifications 03-0005-00-A and 03-0014-00-A, or connecting joints which will meet the critical leak specification of less than 5×10^{-10} std. cc of air per second at the intended use temperature. After assembly, the complete system should be evacuated, baked out at a temperature above the intended use temperature until the pressure rise rate is less than 0.1 micron-liter of gas per gram of alkali metal to be transferred over the anticipated transfer time. The entire system should then be helium leak-checked according to Specification 03-0013-00-A.

A schematic of a generalized alkali metal transfer system for high purity alkali metal is shown in Figure 3.9.3.1. Valves 5, 6 and 10 are angle pattern bellows sealed valves oriented so that they may be easily cleaned back to the seat after contamination with alkali metal. All other valves, except valve 1, are also bellows sealed valves. The pressure relief valve provides protection against inadvertent overpressurization. The molecular sieve trap prevents contamination of the system by back diffusing of oil vapor. It should be baked out under vacuum with valve 8 closed before use. Valve 10 permits the transfer line between valves 5 and 6 to be evacuated and leak checked when the empty container has been used previously and may contain some alkali metal. Valve 11 permits leak checking of the system and may also be used to evacuate the transfer line between valves 5 and 6 through valve 10. The vacuum gauges should be compatible with the ultimate vacuum and pressure rise rate desired. Obviously, they should be ionization gauges, trigger discharge gauges or equivalent if a diffusion pump or getter-ion pump is used and should be backed up with a thermocouple gauge for measuring higher pressures.

The procedures to be used are given in the following paragraphs.

3.9.3.3.1. Leak Checking. Connect a mass spectrometer helium leak detector of adequate sensitivity to valve 11, close valve 11 and leak check the connection according to Specification 03-0013-00-A (Critical Applications). If the empty container is clean, open all valves except 1, 3, 5, 9 and 10, heat all components to between 200° and 250°C, evacuate and leak check according to Specification 03-0013-00-A (Critical Applications). Close valve 11 tightly and disconnect the leak detector.

Valves such as 10 and 11 which may not be leak checked across the seat should be tested before use, while closed and, ideally, should be back filled with argon and capped after disconnecting the leak detector.

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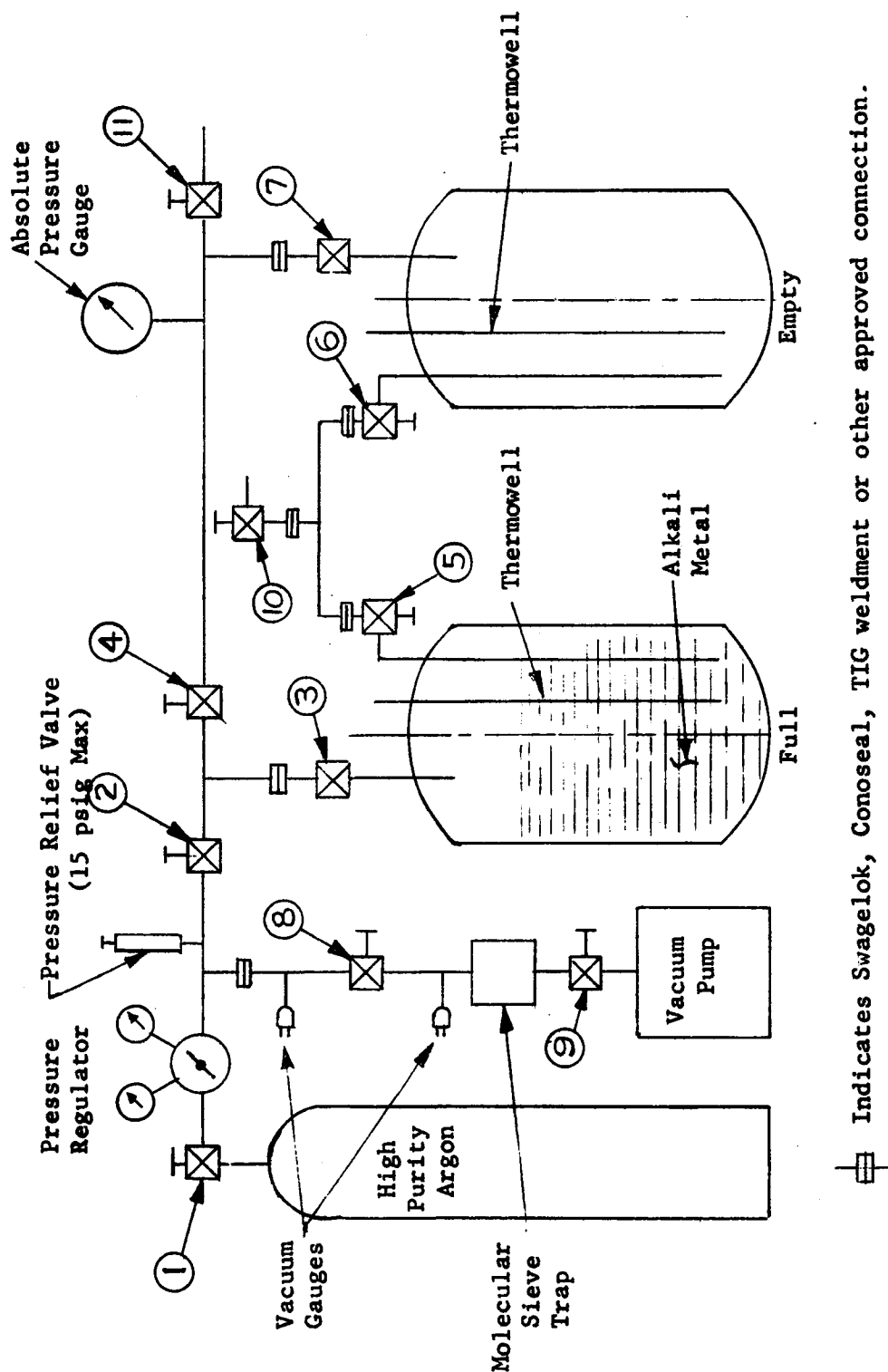


FIGURE 3.9.3.1. Transfer System for Use with High Purity Alkali Metals, Including the High Purity Inert Gas Supply and Vacuum System.

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3.9.3.3.2. Outgassing. Connect valves 10 and 11 and open all valves except 1, 3 and 5. Evacuate through valve 9. Heat all components except valves 1 and 9, the pressure regulator, the pressure relief valve, the molecular sieve trap and the full container to between 200° and 250°C. When pressure has dropped to about 1 micron, close valve 8 and bake out the trap. Cool the trap, open valve 8 and continue bakeout and evacuation until the pressure rise rate, measured with valve 8 closed, is below the maximum allowable rate. Close valve 8, bake out the trap and close valve 9.

3.9.3.3.3. Transfer of Metal. At this point it is assumed that the entire system has been evacuated except for the full container which is presumed to be under a few psig of argon. Heat the full container, the empty container and the transfer line, including valves 5, 6 and 10 to the transfer temperature. Usually the transfer temperature should exceed the melting point of the alkali metal by about 50°F.

Close valves 7, 10 and 11, make sure that valve 8 is also closed, and remove the connection between valves 10 and 11. Valves 3 and 5 are already closed. Close the pressure regulator valve and open valve 1, then set the pressure regulator to give a system pressure of about 25 psia; then open valve 3. This will provide a pressure in the full tank of about 25 psia. If the pressure is too high, bleed down through valve 11 to the atmosphere, or through valves 8 and 9 by using the vacuum system fore-pump.

Close valve 2, then open valve 5 and transfer the desired quantity of alkali metal to the empty container and close valves 5 and 6.

Close the pressure regulator valve and open valves 2 and 7; then open the pressure regulator valve and pressurize both containers to a few psig; then close valves 3, 4, 5, 6, and 7. Cool to room temperature.

Disconnect above valves 5, 6 and 7. Clean valves 5, 6 and 10 immediately according to paragraph 3.10.1 of this specification. The reasons that the valves should be cleaned immediately is that the process is easier before the alkali metal becomes oxidized and that peroxides can form if exposed for long periods. The presence of peroxides is dangerous because they may react explosively with the underlying alkali metal or with the hexane and alcohol used during the cleaning operation.

It is generally easier to discard the transfer line; however, it must be cleaned by using one of the methods described in paragraph 3.10.2 of this specification.

CAUTION: If cesium, rubidium or an alloy like NaK which is liquid at room temperature is involved, the transfer line must be cleared before disconnecting it from the containers. Liquid alkali metals almost always catch fire when exposed to the atmosphere. The heat of reaction of cesium and rubidium with air is usually sufficient to liquify them.

The transfer line may be cleared by evacuating both tanks with valves 3 and 7 open and valves 5 and 6 closed, then closing valves 3 and 7 and blowing down with argon through valves 10 and 11 to clear valves 5 and 6.

3.9.4. Determination of Quantities Transferred

3.9.4.1. Transfers in Air. Rough determinations may be made of the quantity transferred using the inert liquid cover transfer method by weighing the receptacles before and after the transfer; however, an error is involved since the weight of the liquid adhering to the metal remains unknown. Where the surface area to volume ratio is small this error may be insignificant.

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When this method is involved during the chemical analytical techniques, it is generally possible to determine the sample weight chemically.

3.9.4.2. Transfers in Inert Gas and Vacuum Chambers. When making transfers of alkali metals in inert gas and vacuum chambers the quantity transferred may be determined gravimetrically with a balance for solids and liquids, or volumetrically with a burette, hypodermic syringe, volumetric flask, graduated cylinder or some other volumetric device for liquids.

3.9.4.3. Transfers Using Inert Gas Pressure. A number of methods are available for determining the quantity of alkali metal transferred. They are indicated in the following paragraphs.

3.9.4.3.1. Gravimetric. Referring again to Figure 3.9.3.1., either the full or an empty container may be weighed during the transfer operation by placing it on a scale. When this is done the transfer line should be flexible enough to permit the container to move without restraint.

Either container may be weighed before and after the transfer. In this case, one of the volumetric methods described in the next section should be used to estimate the quantity transferred during the transfer operation.

3.9.4.3.2. Volumetric. In all but one of the following methods, it is essential to know the change in internal volume as a function of height for the particular container involved. If it is necessary to know the mass transferred, then the temperature and the change in density with temperature must also be known.

3.9.4.3.2.1. Thermal Gradient Method. This is the simplest of the methods to be described. It consists of determining the vertical thermal gradient of some part of the container which is in contact with the alkali metal and through which heat may be transferred to the environment. Generally this is a thermowell such as is shown in Figure 3.9.3.1. A thermocouple is inserted into the thermowell and a temperature versus depth profile is developed. This profile will show a different rate of change of temperature with depth above and below the alkali metal surface and the point of intersection of the projections of these two lines defines the alkali metal surface.

This method is not applicable when the temperature difference between the alkali metal and the environment is less than about 200°F because the difference in the thermal gradients above and below the surface is not sufficiently great to give a sharp indication of the point of intersection.

3.9.4.3.2.2. Thermal Conduction Methods. This method is simple and is almost universally applicable. It consists of determining the point on some component of the container, which is in contact with both the alkali metal and the space above it, at which the thermal conduction through the wall of the component changes abruptly. For example, if a pipe which is partially filled with alkali metal is heated (preferably in the dark) in the vicinity of the alkali metal-free space interface it will become red hot opposite the free space first and the interface will be indicated by a sharp change between red hot and dark on the pipe wall. The same method may be applied to the outer wall of most containers. Obviously, the method fails when temperature equilibrium is reached and the system is isothermal.

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Another method consists of inserting a small heater or cooler into a well which penetrates the alkali metal surface. A thermocouple is attached to the heater or cooler and makes sliding contact with the well wall. A temperature versus depth profile is developed and the liquid metal surface is defined as the point at which the rate of change of the slope of this curve is a maximum.

Still another method consists of sliding a thermowell containing a thermocouple through a double O-ring sealed feedthrough until contact with liquid is indicated by an abrupt change in temperature. This method is only applicable when an appreciable temperature difference exists between the alkali metal and the environment. It is not recommended for extremely high purity applications.

3.9.4.3.2.3. Electrical Contact Method. This method consists of inserting an electrically conducting probe through a sliding seal which electrically insulates the probe from the metal container until contact with the metal surface is indicated by the fact that the resistance between the probe and container suddenly drops from a very high value to a negligible value. The make point gives a more accurate determination of the level than the break point because surface forces cause the liquid metal to cling to the probe and form a conducting path even after the probe tip is above the liquid metal surface. Double O-ring seals have been used successfully for the insulating sliding seal; however, they are not recommended where the highest purity must be maintained. Stainless steel bellows, of the type used in ultrahigh vacuum valves could be used in high purity systems. The probe, in this case, would be an extension of a high vacuum, ceramic to metal electrical feedthrough. The bellows must be long enough to provide a distance between minimum and maximum extension which is equal to the maximum level change of interest. Also, the bellows should be given adequate support to prevent over-extension or inadvertent contraction during pressure changes in the container; e.g., a screw drive could be used. This method is not applicable when alkali metals may deposit on the insulating region, causing a short.

3.9.4.3.2.4. Electrical Resistance Methods. These methods are based on the change in electrical resistance of some conductor, which penetrates the surface of the liquid metal, as liquid level changes cause more or less of the conductor length to be out of contact with the metal; i.e., a change in resistance as more or less of the conductor is shorted out through the liquid metal to the container. The simplest method consists of measuring the resistance between the metal container and a wire which enters the container through a seal which insulates it from the container. The wire must be long enough to reach a point just above the bottom of the container. Raising or lowering the liquid metal level will then result in a corresponding change in the exposed length of the wire which will, in turn, produce a change in resistance. This technique has three serious disadvantages. The first is that incorporation of a ceramic or glass to metal seal in an alkali metal system limits the operating temperature and pressure because the insulating region may either react with the metal vapors or be rendered conducting by metal deposits. Second, such insulating seals are fragile and may be broken during operation thus exposing the metal to the atmosphere. Third, ideally, the wire should be of small diameter to give a large resistance per unit length; however, the metal may wet the wire and the conduction of the surface layer of liquid metal may have a significant, but unknown, effect on the measured resistance.

3.9.4.3.2.4.1. "J" Type Liquid Level Probe. The disadvantages indicated above which are due to the use of an insulating seal are avoided by the use of the "J" type probe.

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3.9.4.3.2.4.1.1. Description and Method of Use. The complete level measuring system consists of three individual components:

- a. Probe
- b. Control and Monitoring Circuitry
- c. Constant Voltage D.C. Power Supply

A drawing of the probe and a simplified electrical schematic is shown in Figure 3.9.4.1. The probe consists of three concentric conductors separated by ceramic insulating material except at the end which is exposed to liquid metal. At this end, all conductors are welded together in a fashion which completely eliminates exposure of the interior of the probe to liquid metal or to metal vapors. All layers are swaged (before welding the end) to give a tight, evenly supported and easily bendable probe length. After welding the end, the tube package is bent in the form of a "J" and welded into a flange which can be welded directly into the liquid metal container.

Copper leads are attached to the individual conductors at the exposed end, and connections between these leads and the control circuits are made with a total of nine individual conductors as shown in Figure 3.9.4.2.

Current is made to flow through the center tube (Figure 3.9.4.1.) and it passes through the welded tip into the outer tube (sheath) and then through the outer tube into the liquid metal which shorts that part of the tube which is submerged.

The IR drop across the sheath is measured and is proportional to the height of the liquid. The center conductor is used as a voltage lead and carries no current. The probe sheath and center conductor are set up in a bridge circuit so that the temperature-resistivity effects are self compensating. Figure 3.9.4.2. shows the circuit as it actually exists.

A constant D.C. voltage is maintained across points A and B by the power supply, so that the output voltage is proportional only to the height of liquid metal. The output voltage must be measured by a high impedance/null balance instrument to minimize or preferably eliminate loading effects.

The control and monitoring circuit contains a 500-ohm, 10-turn potentiometer which may be used to adjust the signal level. It also contains terminal strips for access to output and input signals.

The D.C. power supply (a Harrison Lab Model 802B has been used) provides excellent load regulation at low voltage and has provision for remote sensing which is necessary to eliminate effects of lead wire voltage drops on voltage across the probe.

3.9.4.3.2.4.1.2. Interconnecting Wiring. Figure 3.9.4.3. shows the recommended wiring between the probe, the control circuit and the power supply. It is essential that all wires to be connected to the probe be grouped together and soldered to the copper leads at the exposed end of the probe at one location. Other types of configurations can be used, but temperature compensation may suffer.

3.9.4.3.2.4.1.3. Calibration. It must be realized that this type of level measuring system is not able to define an absolute level, but will provide a repeatable indication of change in level. Therefore, the probe must be calibrated in place after an initial soaking period at temperatures high enough to insure complete wetting.

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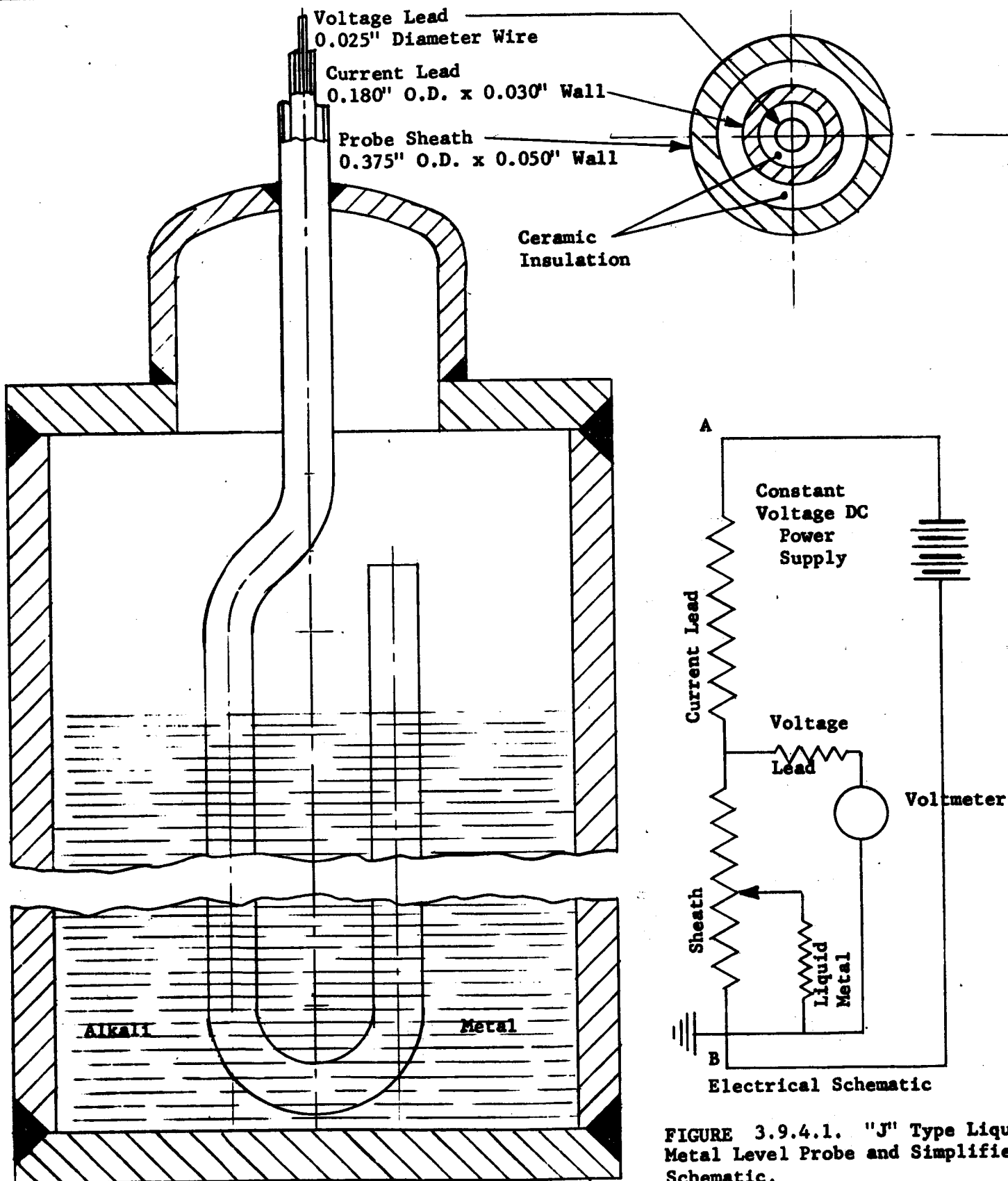


FIGURE 3.9.4.1. "J" Type Liquid Metal Level Probe and Simplified Schematic.

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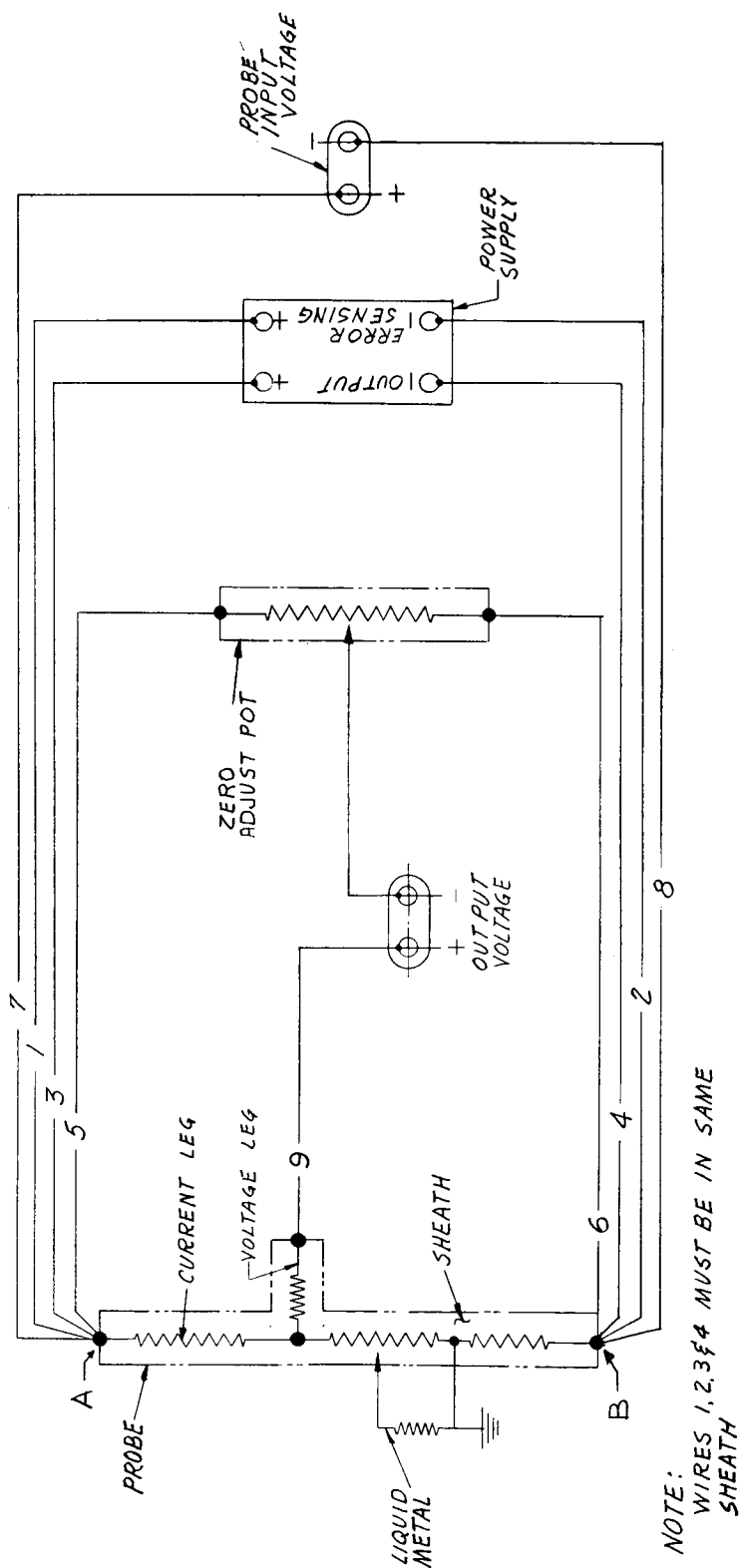


Figure 3.9.4.2. "J" Type Liquid Metal Level Probe - Electrical Schematic.

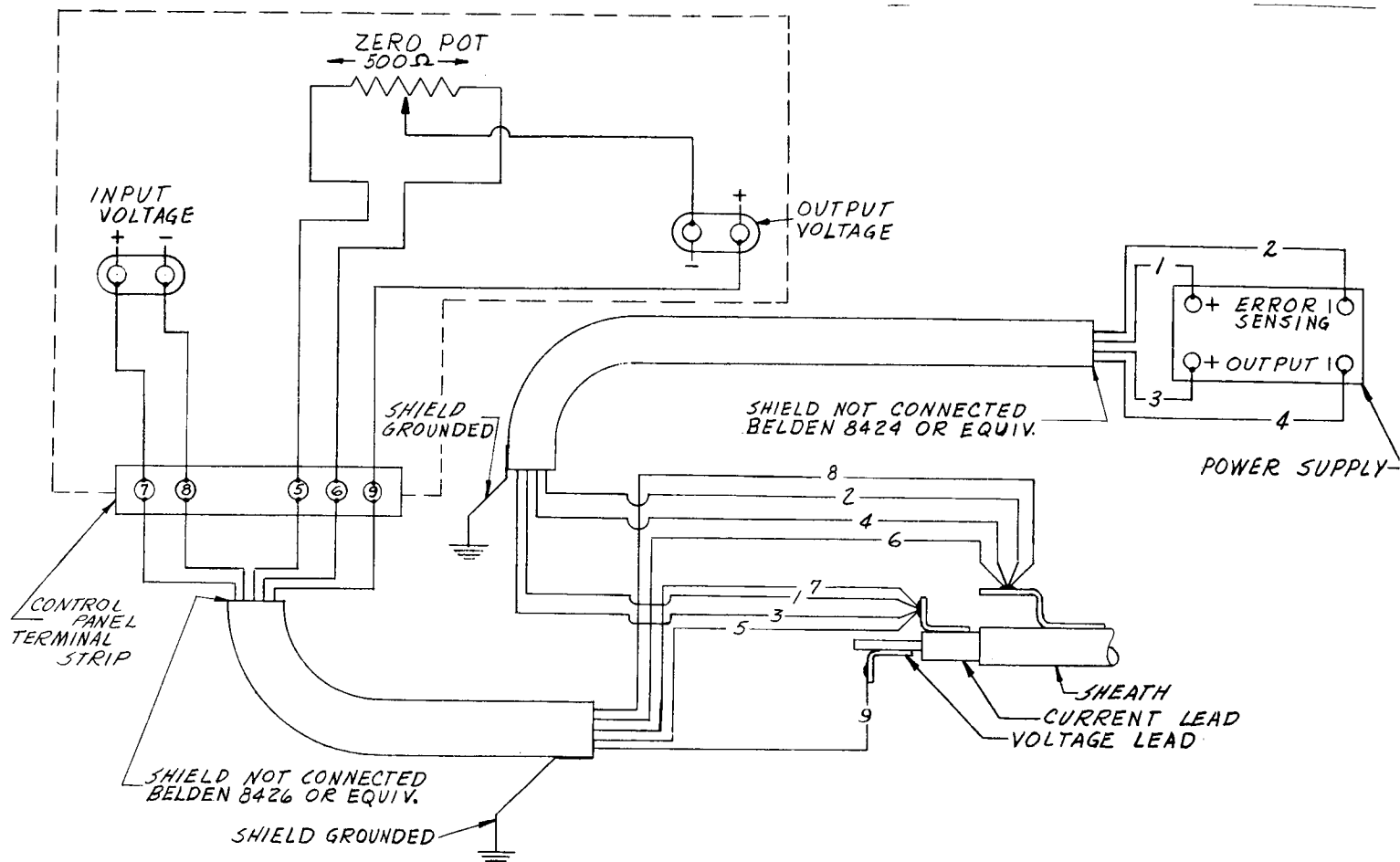


Figure 3.9:4.3. "J" Type Liquid Metal Level Probe - Electrical Schematic.

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The quantity which changes as the level is varied is the effective resistance of the probe sheath as it is shunted by the liquid metal. The calculated resistance of the sheath at 1000°F is 0.000787 ohm per inch of length for the configuration shown in Figure 3.9.4.1. when fabricated from L-605 alloy. Using maximum current of (for the 802B power supply) 1.5 amps gives a theoretical sensitivity of 1.18 millivolts per inch change in level. There are a number of factors which tend to decrease this sensitivity and the following procedure will give an average sensitivity over the length of the probe.

a. After all connections have been made and the probe has been soaked at temperature to insure wetting, adjust the current by means of the power supply controls to 1.4 amps with the tank full of liquid metal (probe completely submerged).

NOTE: The values given below are valid for a 1.4-amp. D.C. current through the probe. They will change if the probe current is increased by means of a bigger power supply.

b. Measure the input voltage at the control box terminals. This value will be approximately 100 millivolts.

c. Adjust the output voltage to a convenient level (approximately 10 millivolts) with the 10-turn potentiometer.

d. Lower the level in the tank until a sharp change in output voltage is noted. This occurs when the level drops beneath the bottom of the probe and the current path is doubled.

e. Raise the level in the tank until the liquid metal just touches the bottom of the probe. The output voltage at this point should be measured after the input voltage has been checked and adjusted to the original value, if necessary, using the power supply controls.

f. The change in voltage over the active length of the probe has now been determined. This should be linear.

g. It is possible to perform this operation in reverse order starting with the liquid metal in contact with the bottom of the probe. However, initial current setting should be somewhat less than 1.4 amps, since it will increase slightly as the level is raised, and it is important that the 1.5-amp rating of the power supply not be exceeded.

Theoretically, the only requirement for complete temperature compensation is that there be no temperature gradient across the cross section of the probe. Axial gradients along the probe are not harmful. However, due to the low level of signals, there are several factors which can cause shifts in output with temperature. Most prominent of these, are thermoelectric emfs between copper and L-605 junctions and liquid metal.

During operation it is necessary to adjust the probe input voltage from time to time to compensate for long term drift characteristics of the power supply. The entire circuit is based on maintaining a constant input voltage at the probe and any change in this voltage is reflected in the output signal.

For purposes of emphasis, it is necessary to measure all voltages with null balance or high impedance instruments to avoid loading effects on the circuit.

The sensitivity is directly proportional to the current through the probe and may be increased many times by using a bigger power supply. The only limit to the current through the probe is the heating of the probe elements. It is estimated that this

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would be insignificant up to 50 amps for the configuration shown in Figure 3.9.4.1. It is also possible to use an A.C. power supply, but practical types of read-out instruments would not be as accurate.

3.9.4.3.2.5. Electrical Induction Methods. The coefficient of self-inductance or simply the self-inductance, L , of a solenoid is a function of the relative permeability, K_m , of its environment. $K_m = \frac{\mu}{\mu_0}$, where μ and μ_0 are the magnetic permeabilities of a material and a vacuum respectively. Consequently, when the environment of a solenoid is changed its self inductance will change. For example, if a small solenoid is placed inside a metal tube which penetrates a liquid metal surface and its self-induction is measured as a function of depth of insertion into the tube, it will be noted that a change in self-induction takes place as the solenoid moves past the liquid metal - free space interface. Such probes must be calibrated in actual liquid metal systems. Furthermore, the tube must be fabricated from a material which is diamagnetic or paramagnetic not ferromagnetic; and if a paramagnetic material is used its magnetic susceptibility should be lower than that of the alkali metal if possible.

Certain manufacturers market liquid metal level indicators which make use of this phenomenon. Mine Safety Appliance Research, Callery, Pennsylvania is one such manufacturer.

3.9.4.3.2.6. Radiation Methods. Beams of gamma rays are attenuated when they pass through material objects, and the amount of attenuation depends directly on the path length and the density of the material. If a radiation source is placed on one side of a liquid metal container and a radiation detector on the other side, the radiation which reaches the detector will be less when the container is full than when it is empty, and with properly positioned detectors it is possible to determine the location of the liquid metal surface.

Certain manufacturers market liquid level indicators which are based on this concept. One such manufacturer is The Ohmart Corporation, Cincinnati, Ohio.

3.9.4.3.2.7. Pressure-Volume Method. This method makes use of the ideal gas law, $PV = nRT$, to calculate a change in volume by determining a change in pressure. For example, if P_1 and V_1 represent the pressure and volume respectively of the gas in a container held at constant temperature and P_2 and V_2 represent the pressure and volume after the container is partially filled with a liquid without removing any of the gas, then nRT is constant and $P_1 V_1 = P_2 V_2$. It is then possible to calculate V_2 if P_1 , V_1 and P_2 are known. The change in volume of the alkali metal in the container is $V_2 - V_1$. Referring to Figure 3.9.3.1., assume that the empty container is partially or completely evacuated after which valve 7 has been closed. Also, assume that the full container has been pressurized to P_1 as indicated on the absolute pressure gauge and that valves 2 and 11 are closed. The initial volume, V_1 , is the free volume in the full container, V_{cl} , plus the volume of the gas manifold between valves 2, 11 and 7, V_{gm} , plus the volume of the pressure gauge, V_{pg} ; i.e., $V_1 = V_{cl} + V_{gm} + V_{pg}$. After some liquid metal has been transferred to the empty container via valves 5 and 6, the pressure in the full container will be P_2 and the free volume

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of this container will now be V_{c2} and $V_2 = V_{c2} + V_{gm} + V_{pg}$. If the gas manifold and full container temperatures are equal and did not change during the transfer, then

$$P_1(V_{c1} + V_{gm} + V_{pg}) = P_2(V_{c2} + V_{gm} + V_{pg}) \quad (1)$$

Since V_{gm} and V_{pg} are both constant, let

$$V_{gm} + V_{pg} = K$$

Then

$$P_1(V_{c1} + K) = P_2(V_{c2} + K)$$

and

$$V_{c2} = \frac{P_1 V_{c1}}{P_2} + \frac{(P_1 - P_2)K}{P_2} \quad (2)$$

Obviously, if K is small with respect to V_{c1} and V_{c2} , the second term on the right of the equation (2) may be neglected. Also, if K is small, the temperature of the inert gas manifold and valves need not be exactly the same as that of the full container.

The same method may, of course, be used to find the quantity of alkali metal added to the empty container.

This is the only volumetric method described which does not depend on a knowledge of the variation of volume with depth for the particular container used in order to calculate the volume of metal transferred.

It should be pointed out that this method may be used to find the free volume in any container. It is only necessary to allow the gas from a known volume to expand into the evacuated unknown volume. The two volumes should be comparable in size for the greatest accuracy.

3.9.4.3.2.8. Measured Volume Method. This method consists of equipping the transfer system with a tank (between valves) which has an accurately known volume, and from which the alkali metal may be displaced completely. This arrangement permits a known volume to be transferred from the tank to the facility. Gas pressure or gravity may be used to effect the transfer.

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3.10. Miscellaneous Operations

3.10.1. Valve Cleaning Procedure. The Hoke HY473A valve is designed to permit complete removal of all alkali metal downstream from the valve seat. The location where the valve is cleaned will be free of water and moist surfaces. The cleaning procedure normally used for solids is as follows:

a. The person cleaning the valve should wear protective clothing, safety glasses, face shield and gloves. The valve MUST be cleaned immediately after use. Do not look into the valve at any time unless protected by a face shield and safety glasses.

b. Using a clean, dry drill bit of the largest diameter which will fit the access tube, remove the bulk of the alkali metal by drilling carefully down to the valve seat by hand. Remove the drill bit from the valve and scrape the metal off of the drill with a screwdriver or spatula (for example) before proceeding further.

c. Remove the metal from the fitting on top of the valve as completely as possible, using a screwdriver or spatula. If the alkali metal starts burning during steps a through c, let it burn itself out.

d. Fill the valve with practical grade hexane if--and only if--the valve is cool and there is no evidence that residual alkali metal is burning.

If a fire should develop during steps d through g, extinguish with a CO₂ fire extinguisher. CAUTION: Do not use a CO₂ fire extinguisher to extinguish alkali metal fires. In steps d through g, the fire will be due to hexane and alcohol which have been ignited by the reaction of the alcohol with very small quantities of residual alkali metal. Throwing sand on such a fire may cause splattering of the flaming hexane-alcohol mixture.

CAUTION: Do not leave bottles of hexane, alcohol or CO₂ fire extinguishers in an area where there is the possibility of an alkali metal fire. For example, do not leave the hexane and alcohol where the valve is. Also, only small quantities of hexane and alcohol are required. About 100 ml of each in separate, LABELED, polyethylene squeeze bottles should be sufficient. Larger quantities should be stored in an area which is cool and free of fire hazards.

Those portions of the cleaning operation which result in the generation of hydrogen by the reaction of ethanol with the alkali metal will be performed in an exhaust hood or other well-ventilated area to avoid accumulation of explosive mixtures of air and hydrogen.

e. Add ethanol to the hexane drop by drop until the hydrogen evolution becomes reasonably vigorous. As the hexane evaporates, add more hexane and alcohol to keep the valve full. Continue adding alcohol until no bubbling is observed in the solution.

f. Remove the hexane-alcohol-ethoxide solution and add fresh hexane and alcohol to the valve. If bubbling occurs, continue adding alcohol until all alkali metal is reacted.

g. Remove the solution from the valve again; an eyedropper is normally used. Add pure alcohol to the valve CAUTIONSLY. Swab the valve interior with cheesecloth until the alcohol and all solid material are removed.

h. Finally, add water to the valve when assured that no free metal remains. Add a few drops of 0.1-1.0 N HCl to the water to neutralize any basic solution.

i. Remove the acid solution from the valve and dry the valve thoroughly.

The cleaning procedure for liquid alkali metals is essentially the same except that the liquid may be poured out or sucked out of the valve rather than drilled out. Moreover, it is the general practice to drain or blow down such valves before disconnecting them from the system.

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3.10.2. General Decontamination and Cleaning Procedures

3.10.2.1. Alkali Metal Containers. Containers such as shipping containers, hot traps, surge tanks, charge pots, still pots and receivers, etc. which contain alkali metals which must be removed may be cleaned by first transferring the bulk of the alkali metal to a disposal drum or shipping container, using one of the techniques described in paragraph 3.9 of this specification, then cutting the container open and completing the cleaning by using one of the following methods.

3.10.2.1.1. Steam Cleaning. This is the fastest method of getting rid of residual alkali metals. It consists of placing the component in a specially constructed room, usually called a "burn room", and then spraying the interior of the component with high pressure steam. The steam reacts with the alkali metal yielding the hydroxide and hydrogen. After conversion, the hydroxide is washed away with water. The air flow through the room is high enough to prevent the overall hydrogen-air ratio from reaching the explosion limits, although locally one may have hydrogen fires or small explosions.

This method is not recommended for delicate parts which may be damaged by the high local temperatures which may develop. Furthermore, it is not generally recommended for metals, such as columbium, which may become hydrogen embrittled; however, if the part is massive enough and the quantity of alkali metal is relatively small, the hydrogen uptake may be insignificant during the short time required for the cleaning operation. Appropriate protective clothing should be worn when applying this method. Stainless steels are subject to stress corrosion cracking when the steam temperature exceeds 150°F.

3.10.2.1.2. Reaction with Alcohols. Residual alkali metals may be removed from containers using alcohols such as ethanol or isopropanol. The part to be cleaned is first filled with hexane, kerosene or some other inert solvent, or it may be placed in another container containing such a solvent. The alcohol is then added slowly with agitation until all of the alkali metal has been converted to the alkoxide. It is preferable to perform this operation under a flowing inert gas cover to minimize the possibility of hydrogen fires and explosions.

When this method is used, it is essential that the alkali metal be freshly exposed so that the surface is not covered with peroxides or superoxides. Such oxides may react violently with organic materials such as the solvents and alcohols used.

This method permits the reaction which produces hydrogen to be controlled so that low temperatures may be maintained. It should be remembered that lithium metal will float on the solvents used, so it is essential that an inert cover gas be used and that the solvent-alcohol mixture be well agitated. The alcohol is best added through a pipe which reaches below the solvent surface in this case.

Fires which occur during the application of this method will generally be solvent fires and may be extinguished with a CO₂ extinguisher. CO₂ should not be used on burning alkali metals since they react to form the oxide and CO.

Appropriate protective clothing should be used while using this cleaning method.

3.10.2.2. Piping, Etc. Pipes, tubes and other similar types of plumbing which contain alkali metal should be drained as much as possible before final cleaning. They may then be cleaned, using techniques similar to those described in paragraphs 3.10.1., 3.10.2.1.1. and 3.10.2.1.2.

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Another technique which may be used for short lengths of pipe, elbows, tees, etc. consists of placing them inside a container which may be evacuated to low pressures (about 10^{-5} torr) and heated to a temperature which is high enough to achieve a reasonable evaporation rate for the alkali metal concerned. The container must contain a condensing surface which operates at a temperature below the freezing point of the metal being distilled. The alkali metal may then be distilled from the component to the condensing surface. The rate at which such distillations take place will be dependent on the conductance of the component and the distillation system, and on the vapor pressure of the alkali metal.

3.10.2.3. Delicate Components. Delicate components should not be steam cleaned except as described below. Instead, the methods described in sections 3.10.1, 3.10.2.1. and 3.10.2.2. should be used. The method selected will depend on the shape, size and materials of construction of the components. It is quite possible that for some components none of these methods will be applicable, and will require the development of a new technique.

Certain components which permit the passage of gas through them may be cleaned by blowing argon or helium through them at a rate sufficient to keep them cool. Then steam or water vapor may be added to the flowing gas stream at a low rate. The inert gas will prevent hydrogen-air explosions by dilution of the hydrogen produced by the reaction of the alkali metal with the water vapor. One disadvantage is that it is difficult to know when all of the alkali metal has been converted to the hydroxide.

3.10.2.4. Vacuum and Inert Gas Chambers. Massive spills of alkali metals inside of vacuum or inert gas chambers may be partially cleaned up by back filling the chamber with an inert gas, opening the chamber and scooping up the metal and placing it in a bucket along with dry sand, then washing off the residual metal with water and finally a wet cloth, providing the residual amount is not too great.

Some large chambers have been equipped with water sprays and drains which may be used to convert the alkali metal to the hydroxide and wash it away under a flowing inert gas cover.

Another method which is especially useful for removing alkali metal surface deposits consists of flowing CO_2 - H_2O vapor mixtures through the chamber. Reactions occur which ultimately produce the carbonate which may be removed by washing away with water.

In the case of chambers that may be heated, the distillation and condensation method described in paragraph 3.10.2.2. may be used to advantage.

ALKALI METAL HANDLING AND CONTROL PROCEDURES CONTINUED

DATE

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3.11. Disposal3.11.1. Disposal Methods

3.11.1.1. Small Quantities. Small quantities of alkali metal such as that remaining in sample tubes are best disposed of by taking them to the "Burn Room" and converting them to the hydroxide with steam. Such quantities should be kept under inert gas cover until they can be transported to the "Burn Room".

Such quantities may also be disposed of by reaction with alcohols in an inert solvent as described in Section 3.10.2.1.2.

3.11.1.2. Large Quantities

3.11.1.2.1. Return to Vendor. Most vendors of alkali metals will dispose of alkali metals returned to them in shipping containers. Generally they make a small charge per pound for this service.

3.11.1.2.2. Burning. Alkali metals may be disposed of by burning in air; however, this requires a sizeable area far removed from human habitation, highways, etc. Such areas are not generally available. Much caustic smoke is produced during the process and this must be prevented from coming into contact with people, animals, houses, machinery, streams, etc.

Smaller quantities may be burned or hydrolyzed in a "Burn Room" which is equipped with a scrubber. The quantity disposed of during a given interval must not exceed the capacity of the scrubber for removing the oxide smoke prior to discharge to the atmosphere.

3.11.1.2.3. Hydrolysis. Large quantities of alkali metal may be disposed of by reaction with water, either in an isolated body of water such as a quarry or at sea. The method consists of opening or puncturing the container before or after throwing it into the water. Generally, a hydrogen explosion will take place in a short time which will tear the container to pieces, thereby bringing all of the alkali metal into contact with the water. The container may be punctured initially after throwing it into the water with a high-powered rifle or a small explosive charge.

Much smoke is generally produced and the explosions are violent, so this operation must be performed at a considerable distance from habitation, public roads, etc. and should only be performed by personnel who are familiar with the hazards involved.

3.11.1.3. Reaction of Alkali Metals with the Atmosphere. Although alkali metals which are exposed to the atmosphere for long periods will eventually be converted to oxides, hydroxides and carbonates, this is not an approved method for their disposal. The reason for this is that most of the alkali metals become covered with peroxides and superoxides. Subsequent handling may bring these higher oxides into contact with the underlying unreacted metal at which time an explosion may take place.

ALKALI METAL HANDLING AND CONTROL PROCEDURES CONTINUED

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4. QUALITY ASSURANCE PROVISIONS. Quality assurance for alkali metals is covered in the individual procurement specifications. These are listed in paragraph 2.2. of this Specification. (Also see paragraph 3.2. of this Specification.)

5. PREPARATION FOR DELIVERY. The preparation for delivery requirements for the alkali metals are covered by the individual procurement specifications.

6. NOTES. None

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED	DATE 15 June 1965	NO. 03-0018-00-A
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APPENDIX AALKALI METAL SURVEILLANCE RECORD

The following form, SP 1061, is to be associated with each lot of alkali metal which is purchased.

SPACE POWER & PROPULSION SECTION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

ALKALI METAL SURVEILLANCE RECORD

WA NO.

SP 11107

REQUESTER

R. A. Fuller

DATE

4/24/65

PHONE

1764

VENDOR

MSAR

INTENDED USE

300 KW Secondary

MATERIAL SPECIFICATION

01-0033-00-B, except that the maximum limit on oxygen is increased to 60ppm K will be hot trapped prior to use.

APPROVED

R. B. Hand

PURIFICATION SPECIFICATION

01-0034-00-B, except that the hot trapping will be performed at 1200°F

APPROVED

R. B. Hand

RECEIVING DATE

5/21/65

MATERIAL DESIGNATION NO.

DRUM D-80

INTRA-PLANT TRANSFER RECORD

	FROM	TO	RECIPIENT	DATE
D	Bldg 200 Dock	Bldg 309 Storage	R. B. Hand	5/22/65
		Bldg 314 Analytical	L. E. Dotson	5/24/65
		Bldg 309 Storage	R. B. Hand	5/26/65
		300 KW Facility	R. A. Fuller	5/30/65
		Bldg 309 Storage	R. B. Hand	6/25/65
		Bldg 200 Dock	G. Van Walde	6/28/65
		Vendor for Disposal	MSAR	6/30/65

ALKALI METAL SURVEILLANCE RECORD - CONTINUED

WA NO.

SP11107

VENDOR ANALYSIS

E

See attached certificate of compliance. Vendor claims that value for lead is due to contaminated analytical reagent.

J. L. King, Buyer

INTERNAL ANALYSIS

INTERSTITIALS

O_2 as $K_2O = 65 \text{ ppm}$
 $C = 3 \text{ ppm}$

ANALYSIS NO.

206

REMARKS

Carbon analysis by Kallman Ledoux and Company — NO 743550, 5/28/65, PO # 030-122697

PERFORMED BY

L. Paian

DATE

5/25/65

F

METALLICS

Na 35	Ca 15	Si 10
Sn 3	Mn < 1	Ti 2
Pb < 1	Cu 5	Al 5
Co 2	Ag < 1	Zn < 10
Fe 10	Cr < 1	Mo < 1

ANALYSIS NO.

304

REMARKS

PERFORMED BY

R. Yoder

DATE

5/27/65

WA NO.

ALKALI METAL SURVEILLANCE RECORD - CONTINUED

REMARKS

Vendor high value fm lead evidently
in error

L. E. Dotson

G

USE APPROVAL

SIGNATURE



DATE

6/29/65

REMARKS

60 ppm O_2 max per 01-0033-00-B is
waived since K will be hot trapped
prior to use.

H

SP 1061 B

ALKALI METAL HANDLING AND CONTROL PROCEDURES - CONTINUED	DATE 15 June 1965	NO. 03-0018-00-A
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APPENDIX BAUTHORIZED ALKALI METAL STORAGE AND USE AREAS -PERMISSIBLE QUANTITIES AND OPERATING CONDITIONS

The following table shows the locations where alkali metals may be used, the maximum quantities permitted, and the extremes of temperature and pressure allowed in each. No other locations are authorized, except that access corridors may be used to transport alkali metals if they are at room temperature.

ALKALI METAL HANDLING AND CONTROL PROCEDURES- CONTINUED

DATE

15 June 1965

NO.

03-0018-00-A

AUTHORIZED ALKALI METAL AREAS

Bldg. No.	Area Designation	Maximum Quant. - lbs.		Operating Conditions	
		In Use	Stored	Press. - psig	Temp. - °F
309	East Bay	1000	2000 (Permanent)	150	2500
	West Bay	1000	1000 (Temporary)	150	2000
	Control Room	None	0.5 (Temporary)	0	Rm. T.
314	North Bay	750	None	10	1500
	South Bay	400	None	150	2500
	Control Room	None	0.5 (Temporary)	0	Rm. T.
318	Facility	3500	None	20	1700
	Control Room	None	0.5 (Temporary)	0	Rm. T.
700	1st Floor - Area A	3	None	165	2500
	1st Floor - Area B	30	None	10	1500
	1st Floor - Area C	0.2	None (Temporary)	0	Rm. T.
	1st Floor - Area D	30	None (Temporary)	10	1600
	1st Floor - Fabrication	20	None	0	300
700	1st Floor - Electric Propulsions Labs	2	None	0	700
	2nd Floor - Physical Test Lab	0.2	None	0	1000
	2nd Floor - Analytical Chemistry Lab	0.5	1	0	300
	2nd Floor - Physical Chemistry Lab	0.5	None	10	1500
	2nd Floor - Ceramic Materials Lab	0.2	None	10	1600



TECHNICAL INFORMATION SERIES

Title Page

AUTHOR R. G. Frank, D. N. Miketta, W. H. Kearns, W. R. Young, and R. B. Hand	SUBJECT CLASSIFICATION	NO. R66SD3007
		DATE Dec. 13, 1965
TITLE MATERIAL AND PROCESS SPECIFICATIONS FOR REFRACTORY ALLOY AND ALKALI METALS		G.E. CLASS I
		GOV. CLASS Unclassified
REPRODUCIBLE COPY FILLED AT (1) MSD LIBRARY, KING OF PRUSSIA, PENNSYLVANIA (2) FPD TECH. INFO. CENTER, CINCINNATI 18, OHIO		NO. PAGES
SUMMARY The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2547. The purpose was the preparation of comprehensive material and process specifications for the procurement, cleaning, and joining of refractory alloys and for the procurement, purification, and handling of the alkali metals. The refractory alloys and alkali metals are to be used in the construction and evaluation of a boiling and condensing potassium corrosion test loop. Although the specifications found herein contain the General Electric Company's designation, anyone who so desires is encouraged to use the contents of the prepared specifications and assign their own specification numbers.		
KEY WORDS		

By cutting out this rectangle and folding on the center line, the above information can be fitted into a standard card file.

INFORMATION PREPARED FOR National Aeronautics and Space Administration under Contract NAS3-2547

TESTS MADE BY _____

COUNTERSIGNED Dr. J. W. Semmel, Jr. DIV. Space Power & Propulsion Section - RSD

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